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Reduced-Pollution Corrosion-Protection Systems

Grumman Aerospace Corp.
Bethpage, NY

Prepared for

Industrial Environmental Research Lab.
Cincinnati, OH

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Coating systems, designed to protect metallic components against corrosive attack using environmentally compatible materials and processes, were evaluated as potential alternatives for their higher polluting counterparts. Viable replacements were established for cyanide cadmium, cyanide copper, and hexavalent chromium electroplating. Alternatives to solvent-borne paints and phenolic-type paint strippers are available with slightly lower performance characteristics than their higher polluting counterparts. Performance characteristics were established for replacement systems of each type through comprehension testing. The performance, economic, and environmental aspects of the new coating systems were compared to those for a currently used control system.

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REDUCED-POLLUTION CORROSION-PROTECTION SYSTEMS

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Final Report
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FOREWORD

When energy and material resources are extracted, processed, converted and used, the related pollutional impacts on our environment and even on our health often require that new and increasingly more efficient pollution control methods be used. The Industrial Environmental Research Laboratory -Cincinnati (IERL-Ci) assists in developing and demonstrating new and improved methodologies that will meet these needs both efficiently and economically.

This report describes the laboratory evaluation of environmentally compatible coating systems for metallic components or potential alternatives to higher polluting counterparts. Several of these coating systems could be used industrially to alleviate environmental pollution. This report should be of value to firms in the aerospace, automotive, building construction, marine equipment, and consumer product industries. The Energy Pollution Control Division is to be contacted for further information on this subject.

David G. Stephan

Director

Industrial Environmental Research Laboratory

Cincinnati

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ABSTRACT

Coating systems, designed for protection of metallic components against corrosive attack using environmentally compatible materials and processes, were evaluated as potential alternatives for their higher polluting counterparts. Viable replacements were established for cyanide cadmium, cyanide copper, and hexavalent chromium electroplating. Alternatives to solvent-borne paints and phenolic-type paint strippers are available with slightly lower performance characteristics than their higher polluting counterparts. Performance characteristics were established for replacement systems of each type through comprehensive testing. The performance, economic, and environmental aspects of the new coating systems were compared to those for a control system currently in use.

Alternative coating systems evaluated for cyanide cadmium electroplating included non-cyanide cadmium electroplating, mechanical plating of cadmium and tin-cadmium, spray-and-bake aluminum-filled resin coatings, and ion-vapor-deposition (IVD) of aluminum. Each of these alternatives eliminated the cyanide waste treatment problem while the last two types also eliminated the use of cadmium, another toxic material. Although no system can be considered the best alternative to cyanide cadmium plating, each system has certain advantages while offering the same basic performance as cyanide cadmium plating. Non-cyanide copper electroplating (the alternative evaluated for cyanide copper electroplating) and trivalent chromium electroplating (the alternative to hexavalent chromium electroplating) were shown to provide performances comparable to their higher polluting, higher waste treatment requirement control systems.

Potential alternatives evaluated for solvent-borne paints included water-borne paints and powder coatings, both of which eliminate the need for solvent collection systems in coating applications. Although performance characteristics of both water-borne paints and powder coatings were shown to be comparable to those for the solvent-borne control system, no single-system provided equivalent performance for all characteristics.

Evaluation of non-phenolic paint strippers showed the availability of potential alternatives to phenolic strippers that would eliminate the phenolic waste disposal problem. Acid and non-acid immersion and brush-on type strippers were evaluated.

This report was submitted in fulfillment of Project R804331 by Grumman Aerospace Corporation under the (partial) sponsorship of the Office of Research and Development, U.S. Environmental Protection Agency. This report covers the period from 1 August 1976 to 31 December 1979. Long-term outdoor exposure testing was completed on 15 July 1980.

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SECTION 1

INTRODUCTION

Mounting concern over environmental pollution and more stringent EPA and OSHA regulations have exerted pressure on the metal-finishing industry to expand use of new less-polluting corrosion protection systems in lieu of currently used organic solvent systems and cyanide- or cadmium-containing plating solutions. The aerospace and metal-finishing industries have available many new coating systems that can be considered non-polluting or as having reduced-pollution characteristics compared to the currently used systems. Corrosion-resistance data for these systems are limited, however, especially with respect to long-term exposure. As a result, customer confidence in these systems is not high and has inhibited their widespread utilization. The metal-finishing industry is especially resistant to the introduction of new systems for which supporting test data are limited and manufacturing costs are not clearly defined.

This project was initiated, therefore, to evaluate less-polluting corrosion protection systems as viable alternatives to presently used higher pollution systems. The approach taken was to evaluate and demonstrate the durability of the new systems by conducting extensive tests which serve as acceptance criteria by the aerospace and metal-finishing industries.

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SECTION 2

CONCLUSIONS

Several replacement systems are available for cyanide cadmium electroplating, including non-cyanide cadmium electroplating, mechanical plating, spray-and-bake aluminum coating, and ion-vapor-deposited (IVD) aluminum. These systems, each of which has advantages and disadvantages compared to conventional cadmium electroplating, can provide protection equivalent to that given by cyanide cadmium electroplating, for various applications and cause less environmental effects than cyanide cadmium electroplating. These systems are summarized below:

- Kadizid, the selected non-cyanide cadmium electroplating system from Lea Ronal, Inc., provides excellent adhesion and corrosion resistance with no sign of hydrogen embrittlement while eliminating the need for cyanide waste treatment

- Transiflo, a mechanically plated cadmium coating from Minnesota Mining and Manufacturing Company, provides good adhesion and excellent corrosion resistance with no sign of hydrogen embrittlement. No waste treatment is required for this plating system

- Alumazite Z, a spray-and-bake aluminum coating from Tiodize Co., Inc., provides excellent adhesion and corrosion resistance with no sign of hydrogen embrittlement. Since no cyanides or cadmium are required, the associated waste treatment problems are eliminated

- Ivadize, an ion-vapor-deposited (IVD) aluminum coating from McDonnell Aircraft Corporation, provides excellent adhesion and corrosion resistance with no potential for hydrogen embrittlement. Environmental and waste treatment problems do not exist because cyanide or cadmium is not used in the process.

Water-borne paints and powder coatings are available which provide performance conforming to the requirements of MIL-C-81773 (polyurethane topcoat). The hazards associated with the use of solvent-borne paints, as well as the need for solvent collection facilities, are eliminated through the use of either of these replacement coatings:

- Three of the water-borne paints evaluated, including one air-drying system and two bake-curing systems, have coating properties close to those for the control system. These include film properties (appearance, adhesion, impact, and flexibility), fluid resistance, and weatherability

- Five of the powder coatings evaluated also have coating properties similar to those of the control system. These include film properties (appearance, adhesion, impact, and flexibility), fluid resistance, and weatherability.

A non-cyanide copper electroplating system is available which is capable of performance equivalent to that of currently used cyanide-type copper electroplating systems per MIL-C-14550. Enthone's Enthobrite Cu-942 provides excellent adhesion, solderability, and decarb protection with no sign of hydrogen embrittlement while eliminating the need for cyanide waste treatment.

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A trivalent chromium plating system, evaluated as a replacement for decorative hexavalent chromium electroplating, provided performance equivalent to that for the hexavalent system while eliminating the toxicity and waste treatment problems associated with hexavalent chromium. Trichrome, from the Harshaw Company, Cleveland, Ohio, showed good appearance, adhesion, and corrosion resistance, with no sign of hydrogen embrittlement.

Several acid and non-acid, brush-on and immersion-type, non-phenolic paint strippers show promise as potential replacements for phenolic paint strippers. The performance of these systems approximates that of the standard phenolic paint stripper (MIL-R-81294) in appearance, removal power, rinsability, coating and remover residue, and refinishability.

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SECTION 3

RECOMMENDATIONS

This technology development program established that many new less-polluting corrosion protection systems are available that are viable alternatives to present high-pollution systems. Results obtained have made it abundantly clear that this program should be continued since this effort has spurred manufacturers to develop or improve less-polluting metal-finishing systems.

If the development efforts were continued, aerospace and metal-finishing firms would be able to implement the following new systems with a high degree of confidence:

- Water-borne, chemical milling maskant systems evaluated under the present grant have shown promise as replacements for high-polluting systems. Additional pilot-plant (50-gallon) testing on typical flat and curved panels should be conducted to demonstrate the viability of these systems for future and near-future commercial and military aircraft applications. This testing would provide the data needed to implement the less polluting, water-borne maskants within the critical timeframe on such new aircraft as the 757 and 767 commercial airliners and ATF, FSW, V/STOL, AV-8B and F-18 military aircraft.

- Several manufacturers of water-borne paints, paint strippers, and spray-and-bake corrosion protection coatings are developing new products, or improving existing products, as a direct results of the favorable publicity that this program has received. These companies would like to have these new systems evaluated.

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SECTION 4

REPLACEMENT COATINGS FOR CYANIDE CADMIUM

APPROACH

This phase of the program was directed toward evaluation of potential replacement systems for cyanide cadmium plating. The approach taken involved the evaluation of several non-cyanide cadmium electroplating systems, two spray-and-bake aluminum coatings, ion-vapor-deposited aluminum, mechanically plated cadmium and tin-cadmium, a resin topcoat for several of these systems, and selection of the best system of each type.

STUDY AREAS

Evaluation of the replacement coatings for cyanide cadmium plating involved the following:

- Selection of substrates, pretreatment procedures, and application procedures
- Testing and evaluation of candidate coatings
- Economic and environmental evaluation.

BACKGROUND

Cadmium is normally plated from cyanide-type electroplating baths. The high level of toxicity of the cyanides, however, necessitates that costly waste treatment procedures using either chlorine or hypochlorites be used to destroy the cyanide when these solutions are dumped (Ref. 1). Several cyanide-free cadmium electroplating baths are available; preliminary investigations indicated that these both have potential as replacement systems. Mechanical plating of powdered cadmium also provides a potential alternative for cyanide-type cadmium solution electroplating. Several aluminum coatings are also available as potential replacements for cadmium plating. Ion-vapor-deposited (IVD) aluminum and aluminum-filled resin spray-and-bake coatings are of particular interest. Use of aluminum coatings would eliminate the toxicity problems related to the use of cadmium. The systems that were considered for potential replacement of cyanide cadmium plating are shown in Table 1.

SUBSTRATE PRETREATMENT AND COATING APPLICATION

Substrates that are typically cadmium-plated include 4340, 4130, D6AC, and 300M high-strength steels. The principal substrates chosen for the various types of replacement coatings were:

- Non-cyanide cadmium - 4130, 4340
- Mechanical plating - D6AC, 4340, 300M
- Spray-and-bake aluminum - 4130

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TABLE 1. POTENTIAL REPLACEMENT SYSTEMS FOR CYANIDE CADMIUM ELECTROPLATING

Type	Manufacturer	System
Non-cyanide cadmium	Lea Ronal	Kadizid
	Lea Ronal	Cad-Al
	Enthone	Enthobrite Cad-935
Mechanical plating	3M	Transiflo
Spray-and-bake aluminum	Tiodize	Alumazite Z
	Hi Shear	Hi Kote 3
Ion-vapor-deposited (IVD) aluminum	McDonnell Aircraft	Ivadize
Resin topcoat (for above systems)	Empire	Empigard I

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- IVD aluminum - 4340.

Pretreatment of the substrates for non-cyanide cadmium plating was similar to the procedure used for cyanide cadmium plating: solvent-clean, alkaline-clean, and vapor-blast. Pretreatment of substrates for mechanical plating, which included vapor degreasing and hot alkaline cleaning, was required to provide an adherent coating. Pretreatment for the spray-and-bake aluminum coatings included vapor degreasing, dry honing, and force-filtered air blow-off. The IVD aluminum pretreatment included alkaline cleaning and acid deoxidizing.

The non-cyanide cadmium plating systems were prepared according to the manufacturers' recommended procedures. Panels were mechanically plated by 3M Company according to their standard procedure. The spray-and-bake aluminum coatings were applied according to manufacturers' recommended procedure (Alumazite Z) with and without an epoxy primer and by the manufacturer (Hi Kote 3) with and without primer. Application of IVD aluminum was accomplished according to standard procedure by McDonnell Aircraft Corporation.

TESTING AND EVALUATION OF CYANIDE CADMIUM REPLACEMENT COATINGS

The four types of potential replacement coatings for cyanide cadmium plating, i.e., non cyanide cadmium, mechanical plating, spray-and-bake aluminum, and IVD aluminum, were tested to determine the best system available in each category and to aid in evaluating the relative merits of the four types.

Non-Cyanide Cadmium Plating

Three non-cyanide cadmium plating systems were evaluated as potential replacements for cyanide cadmium electroplating. These systems, Lea-Ronal's Kadizid, Lea-Ronal's Cad-Al, and Enthone's Enthobrite Cad-935, were evaluated in Hull cell tests to determine throwing power and

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brightness. Zinc-plated Hull cell panels were stripped of zinc in 50% HC1, rinsed, dipped in 20% H_2SO_4, rinsed, and plated for 2 min at 3A in a 267-ml (0.07 gal) Hull cell. A pure cadmium anode was used. Kadizid showed the greatest bright range and thickest deposit in these tests (Table 2). The appearance of the Kadizid plate was also better than that of the other non-cyanide systems, including the cyanide-control system. Lea-Ronal's Cad-Al showed a thin, semi-bright deposit with a narrow bright range. The plate produced by the Enthobrite Cad-935 system was discolored; the discoloration worsened on standing. Both of these systems were eliminated from further testing. Kadizid was selected for optimization and final characterization tests.

Further testing of the Kadizid system was done in a 2-l (0.53 gal) bath. Throwing power, as determined by the bent cathode test, was excellent (Table 3). Hull cell panels, bent as shown in Table 3, were stripped of zinc and plated for 15 min at varying current densities. The plated panels were examined for thickness and deposit quality. The leveling power of the Kadizid system was determined by plating an etched panel and examining the panel for surface smoothness. Hull cell panels were etched in 50% by volume HCl for one minute. One panel was then plated for 15 min at 270 A/m^4 (25 A/ft^2). The result was a smooth bright finish (Fig. 1), showing the good leveling power of the Kadizid system.

The adhesion of the Kadizid plate was excellent. 4340 steel coupons, 2.5 cm by 10.16 cm (1 in. by 4 in.), were plated to 15.2 to 22.8 μ m (0.6 to 0.9 mil) in Kadizid (540 A/m^2 or 50 A/ft^2 for 20 min), baked at 190 ± 14°C (375 ± 25°F) for 3 hr, dipped in 2% H_2SO_4 to re-activate the surface, and dipped in Enthox chromate (chromate conversion coating, Type II). These panels were subjected to the bend test and showed no evidence of peeling or flaking. Panels that were cyanide cadmium-plated also showed excellent adhesion. Non-cyanide cadmium-plated test panels are shown in Fig. 2.

The Kadizid system was subjected to final characterization tests and compared to other cadmium replacement systems (see the following section).

Mechanical Plating

Cadmium and tin-cadmium applied by mechanical plating were also considered as potential replacement systems for cyanide cadmium electroplating. The 3M Company's Transiflo process was used to apply these coatings.

Cadmium was applied to 4340 and D6AC steel panels to a thickness of 20.4 μm (0.3 to 0.8 mil), after which a chrome conversion coating was applied. A 50-50 mixture of tin and cadmium was applied to the 4340 and D6AC steel panels to a thickness of 5.1 to 17.8 μm (0.2 to 0.7 mil); no conversion coating was applied. The evaluation of these coatings was based on adhesion and corrosion resistance tests. Bend tests indicated that the adhesion of both the cadmium and tin-cadmium mechanically plated coatings is satisfactory (Table 4). Mechanically plated cadmium and tin-cadmium test panels are shown in Fig. 3 and 4, respectively. Corrosion testing results are discussed in the following section.

Spray-and-Bake Aluminum

Two spray-and-bake aluminum coatings were evaluated as potential replacements for cyanide cadmium plating. Alumazite Z from Tiodize and Hikote 3 from Hi Shear were applied to 4130 steel panels with and without MIL-P-23377 epoxy primer. The coatings were tested to determine their adhesion, corrosion, and fluid resistance. The adhesion of both coatings was excellent, with and without epoxy primer (Table 5). The corrosion resistance of Alumazite Z without primer was poor; each panel failed after 96 hr in salt spray due to red rust. With the epoxy primer, corrosion resistance was extended significantly (see the following section). Corrosion resistance of the Hikote 3 coating was about

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TABLE 2. CADMIUM PLATING - HULL CELL TEST

Plating System	Bright Range, A/m^2 (A/ft^2)	Deposition Rate, µM/Min (Mils/Min) at 430A/m^2 (40 A/ft^2)	Plate Thickness*, µM (Mils)	Appearance
Enthobrite Cyanide Cadmium	54-430 (5-40)	0.76 (0.03)	1.52 (0.06)	Burnt deposit at higher current densities
Kadizid	54-1075 (5-100)	0.64 (0.025)	1.27 (0.05)	Bright
Cad-Al	54-540 (5-50)	0.25 (0.01)	0.51 (0.02)	Semi-bright
Enthobrite Cad-935	22-1190 (2-110)	0.51 (0.02)	1.02 (0.04)	Discolored

*Hull cell test run at 3 A for 2 min

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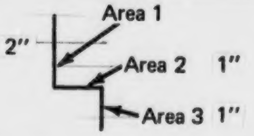
TABLE 3. KADIZID NON-CYANIDE CADMIUM PLATING/THROWING POWER - BENT CATHODE TEST

Current Density, A/m^2 (A/ft^2)	Time, Min	Plate Thickness, µM (Mils) Area 1*	Area 2*	Area 3*	Comments
86 (8)	15	1.02 (0.04)	1.02 (0.04)	2.03 (0.08)	Excellent
194 (18)	15	3.05 (0.12)	3.05 (0.12)	8.38 (0.33)	Excellent
302 (28)	15	7.11 (0.28)	4.32 (0.17)	12.7 (0.50)	Excellent
410 (38)	15	9.14 (0.36)	9.14 (0.36)	14.5 (0.57)	Pitting in area 2
518 (48)	15	13.5 (0.53)	12.4 (0.49)	19.1 (0.75)	Pitting over entire surface, streaking in area 3

*Where



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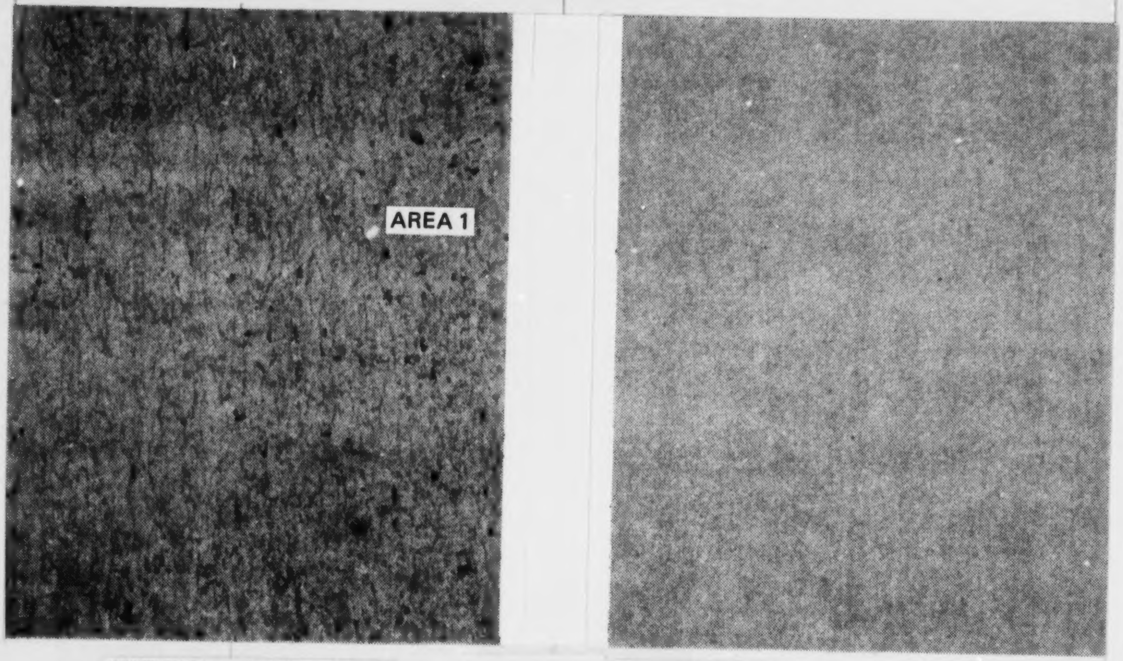

UNPLATED PANEL* (100X)

PLATED PANEL* (100X) 270A/M^2 (25 A/FT^2), 15 MIN, 10.4 μM (0.41 MIL) PLATE

*PANELS ETCHED BY ONE-MINUTE SOAK IN 50% BY VOLUME HCℓ

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Figure 1. Kadizid Non-Cyanide Cadmium Plating - Leveling Power Test.

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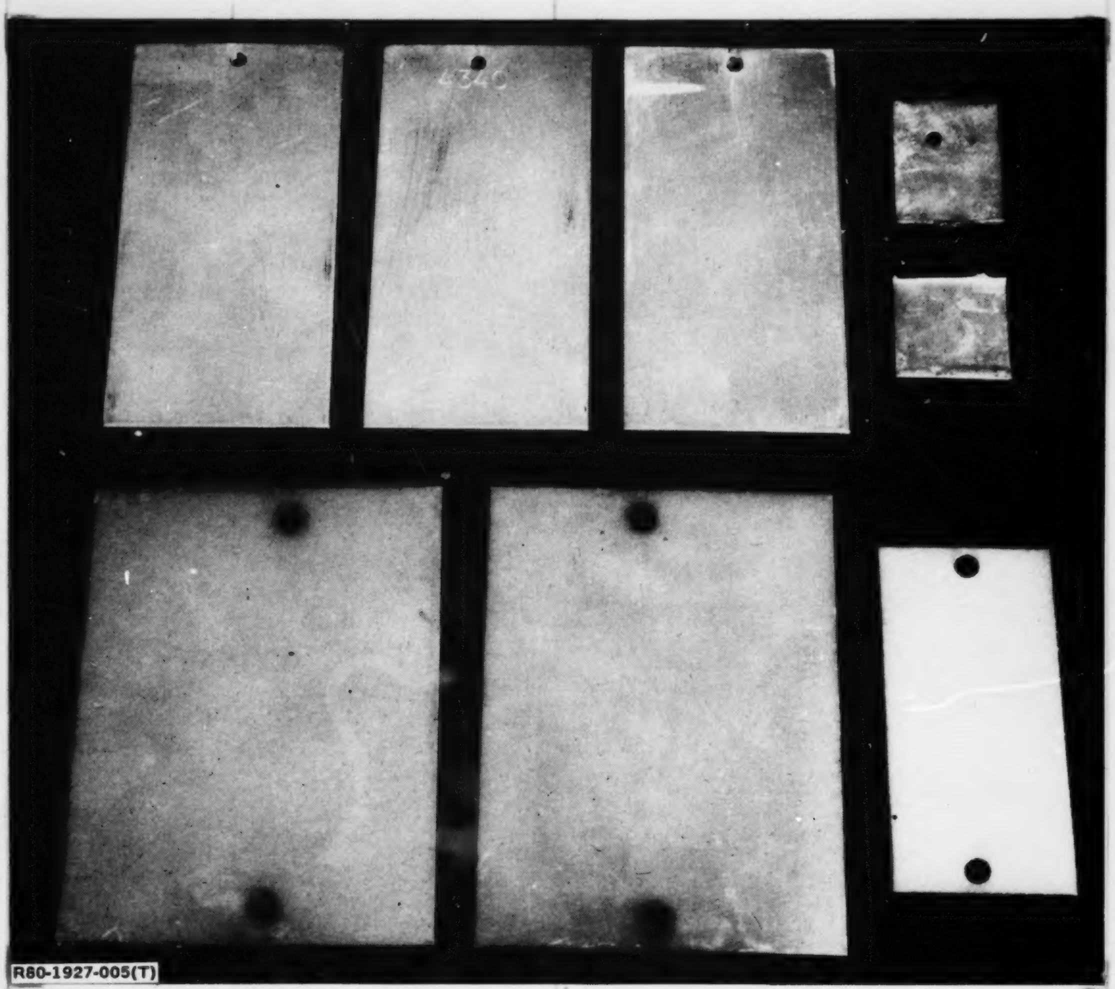

Figure 2. Non-Cyanide Cadmium-Plated Test Panels.

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TABLE 4. MECHANICAL PLATING - PRELIMINARY EVALUATION

Property	Mechanically Plated		Cyanide Cadmium Electroplate (With Chromate Conversion Coating)
	Cadmium (With Chromate Conversion Coating)	Tin Cadmium	
Appearance	Scratched	Scratched	Smooth
Adhesion	Pass	Pass	Pass
Corrosion resistance, hr to failure in 5% salt spray			
Substrate — D6AC	Pass 1000	516	Pass 1000
4340	2 Pass 1000 2 Fail @ 624	558	Pass 1000
Thickness, μm (mil)	7.6 — 20.4 (0.3 - 0.8)	5.1 - 17.8 (0.2 - 0.7)	3.8 - 8.9 (0.15 - 0.35)

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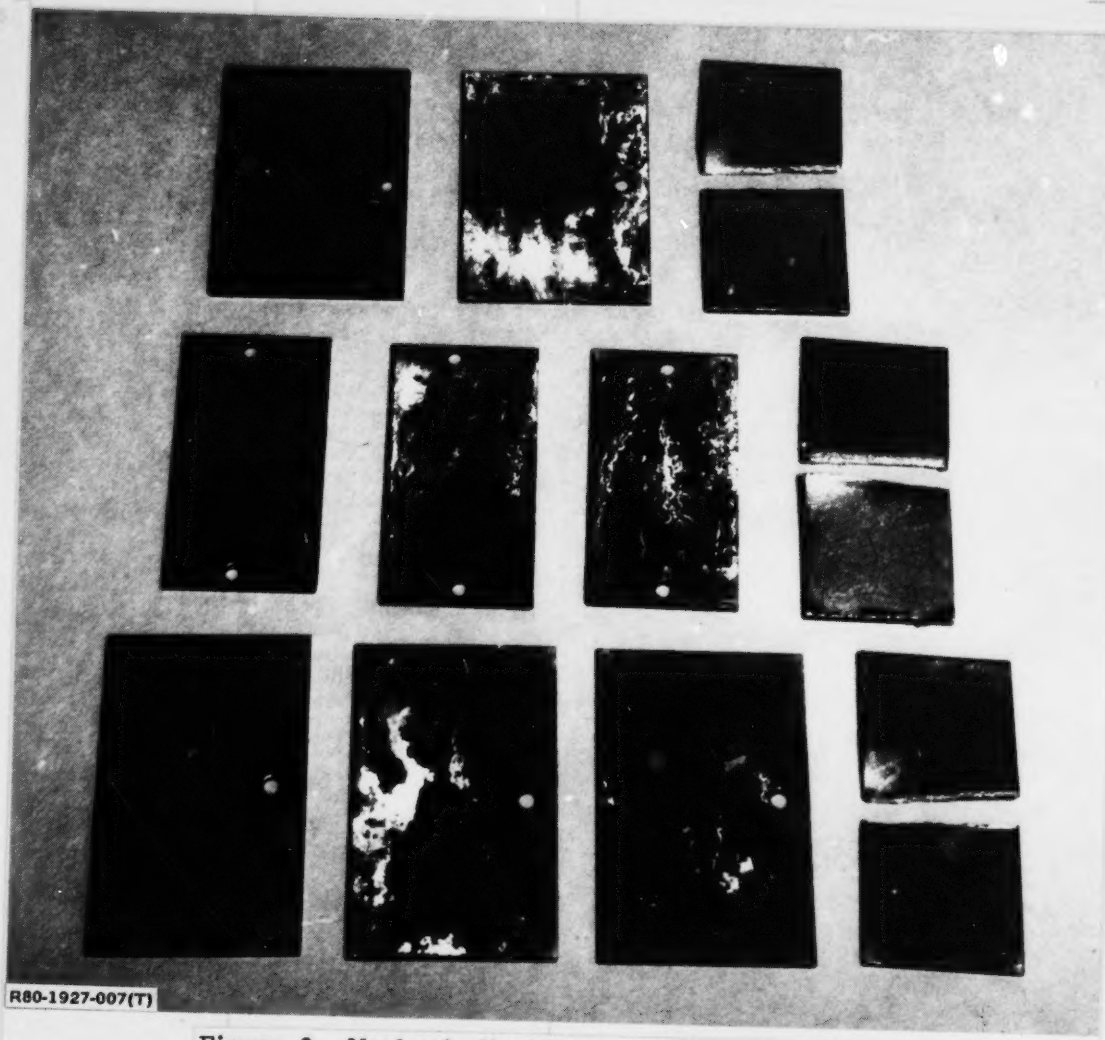

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Figure 3. Mechanically Plated Cadmium Test Panels.

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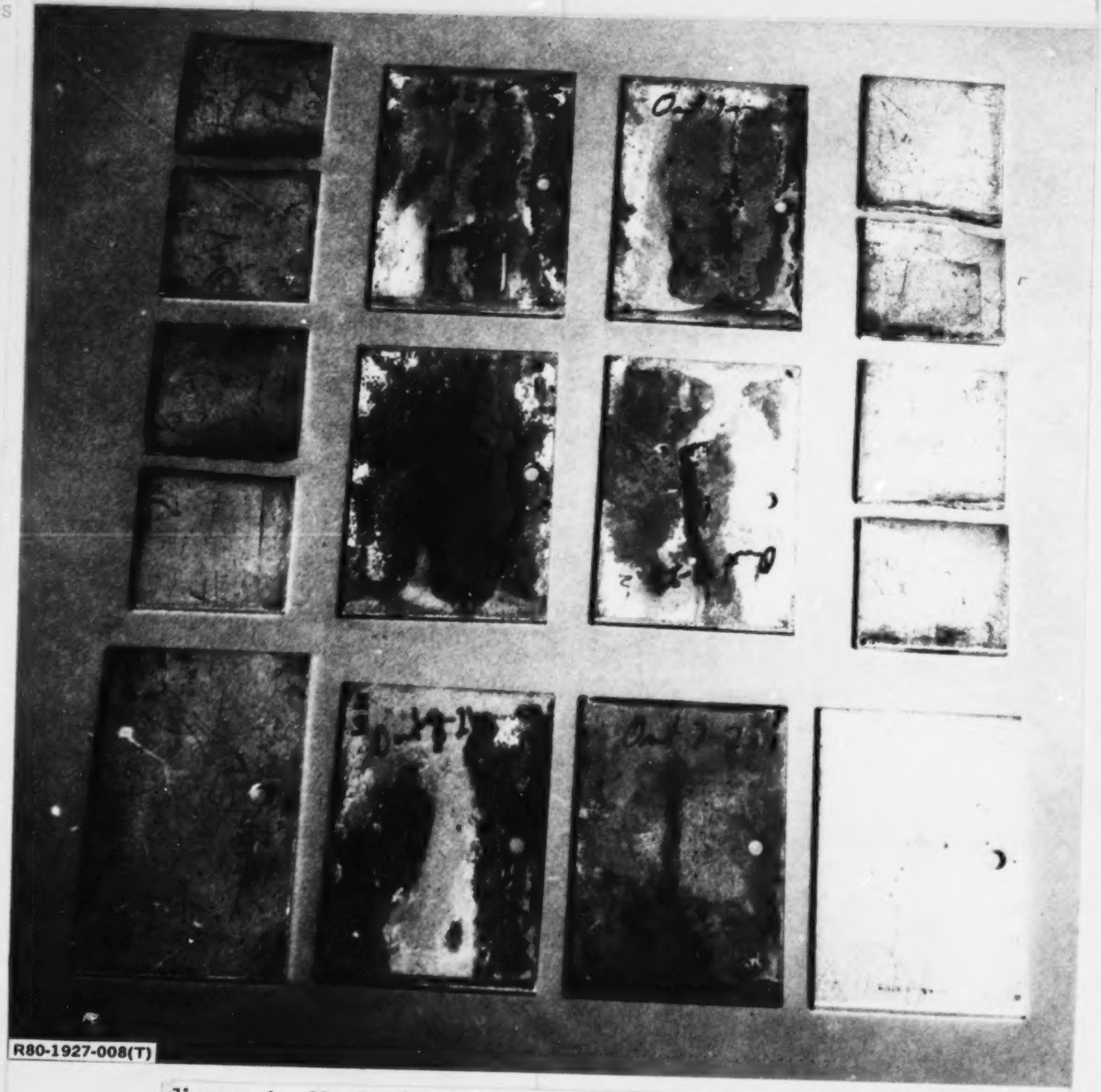
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Figure 4. Mechanically Plated Tin-Cadmium Test Panels.

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TABLE 5. SPRAY-AND-BAKE ALUMINUM - PRELIMINARY EVALUATION

Property	Tiodize Alumazite Z No Primer	Tiodize Alumazite Z Epoxy Primer	Hi-Shear Hi-Kote 3 No Primer	Hi-Shear Hi-Kote 3 Epoxy Primer
Adhesion – bend test	No lifting or peeling	No lifting or peeling	No lifting or peeling	No lifting or peeling
Corrosion – salt spray	96 hr to red rust*	700 hr to red rust*	300 hr to red rust**	200 hr to red rust**
Fluid resistance (R.T.)				
– Skydrol 500	–	Heavy white ppt @ 650 hr***	White ppt @ 96 hr Good adhesion @ 4000 hr	–
– 5606 Hydraulic fluid	–	No effect @ 4000 hr	No effect @ 4000 hr	
– Diester oil	–	No effect @ 4000 hr	No effect @ 4000 hr	
Thickness, um(mil)	5.1 - 12.7 (0.2 - 0.5)	5.1 - 12.7 (0.2 - 0.5)	10.2 - 12.7 (0.4 - 0.5)	10.2 - 12.7 (0.4 - 0.5)
Hydrogen embrittlement		Pass 200 hr @ 75% unts**** (3 specimens)	–	–

* Average of 2 panels

** Average of 4 panels

*** Loss of adhesion occurred prior to 4000 hr

****Ultimate notched tensite strength

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the same with and without epoxy primer. Both coatings withstood extended exposure to aircraft fluids. Hydraulic fluid 5606 and diester oil had no effect on either coating during a 4000-hr exposure. A white precipitate formed during the Skydrol 500 exposure period; loss of adhesion of the Alumazite Z resulted from the exposure. The Hikote 3 coating showed good adhesion after 4000 hr in Skydrol 500. Figures 5 and 6 show the spray-and-bake aluminum-coated test panels.

Ion-Vapor-Deposited (IVD) Aluminum

Panels of 4130 steel were coated with aluminum by McDonnell Douglas using their ion-vapor-deposition (IVD) process. A chromate conversion coating was applied to the coated panels. Adhesion of the IVD aluminum coating, as determined by the bend test, is excellent (Table 6). Coated test panels are shown in Fig. 7. Corrosion resistance of this coating is also excellent (see the following section).

Coating Corrosion Resistance

The relative corrosion resistance of each of the coating systems was determined using the 5% salt spray corrosion test. Two to four panels of each system were subjected to 5% salt spray until failure. Cyanide cadmium-plated panels were also run as controls. The corrosion resistance of a coating system, measured as time to failure, varies with the substrate to which it is applied. For example, the average life in salt spray of cyanide cadmium-plated 4340 steel panels was 2500 hr, while that for similarly coated 4130 steel panels was only 575 hr (Table 7). Cyanide cadmium-plated test panels are shown in Fig. 8.

Non-cyanide cadmium plating and mechanically plated cadmium, both with chromate conversion coating, showed corrosion resistance approximately equivalent to the cyanide cadmium-plated control on 4340 and D6AC steels. While a wide spread in the data is evident, the potential for very good corrosion protection is available with these coatings (see Table 7). The mechanically plated tin-cadmium, which was not chromate-conversion-coated, did not last as long in 5% salt spray as the cyanide cadmium control coating. It did, however, exceed 450 hr for all specimens, which is well over the minimum requirement specified for corrosion resistance in MIL-C-81562, "Coatings, Cadmium, Tin-Cadmium and Zinc (Mechanically Deposited)."

The spray-and-bake aluminum coatings were applied to 4130 steel panels and tested to failure in 5% salt spray. The corrosion resistance of the Alumazite Z with epoxy primer was equivalent to that of the cyanide cadmium coating with chromate conversion coating on 4130 steel panels (Ref. Table 7). Although the Hikote 3 coating is less resistant than the control, it did exceed the 96-hr minimum requirement for cadmium plating (QQ-P-416, "Plating, Cadmium (Electro-deposited)").

Test panels of 4130 steel coated with MIL-P-23377 primer and Alumazite Z spray-and-bake coating were exposed at Grumman's Fire Island Test Site for 3, 6, 9, and 12 months. No sign of corrosion on the specimens was observed after exposure. Sheet bend tests conducted on the exposed specimens showed that the coating had good adhesion with little or no peeling.

The IVD aluminum coating provided excellent corrosion resistance for 4130 steel. Each of the IVD coated panels lasted significantly longer in 5% salt spray than did the cyanide cadmium control panel (Ref. Table 7).

Empigard I, a thin resin coating used to extend the corrosion resistance of plated material, was also evaluated in 5% salt spray corrosion. This coating was applied to panels coated with several of the candidate cyanide cadmium replacement coatings, including mechanically plated cadmium and tin-cadmium and the Hikote 3 spray-and-bake coating. The Empigard I coating somewhat extended the corrosion resistance of the tin-cadmium mechanically plated 4340 steel panels (Ref. Table 7), but had

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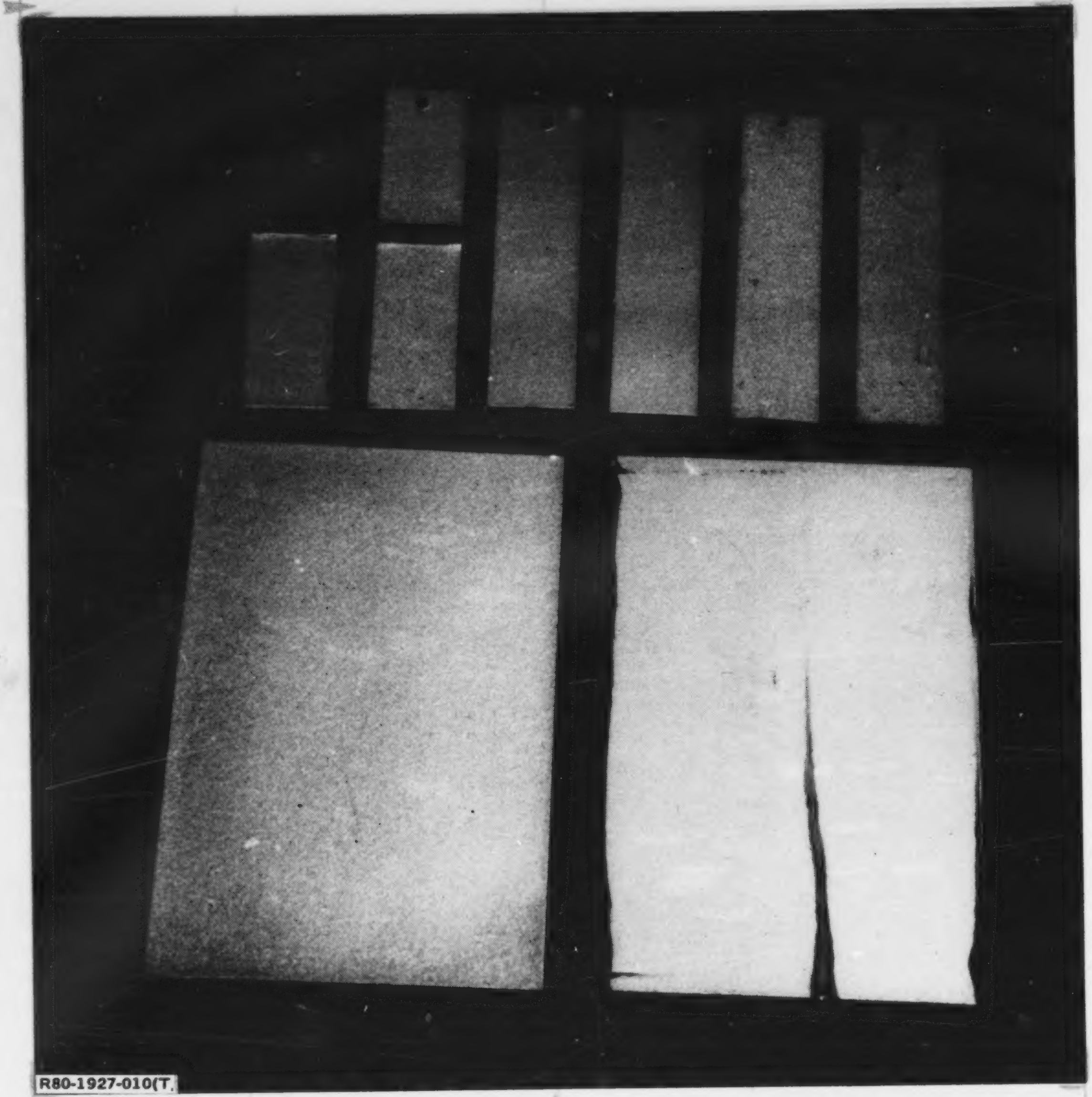

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Figure 5. Spray-and-Bake Aluminum - Alumazite Z-Coated Test Panels.

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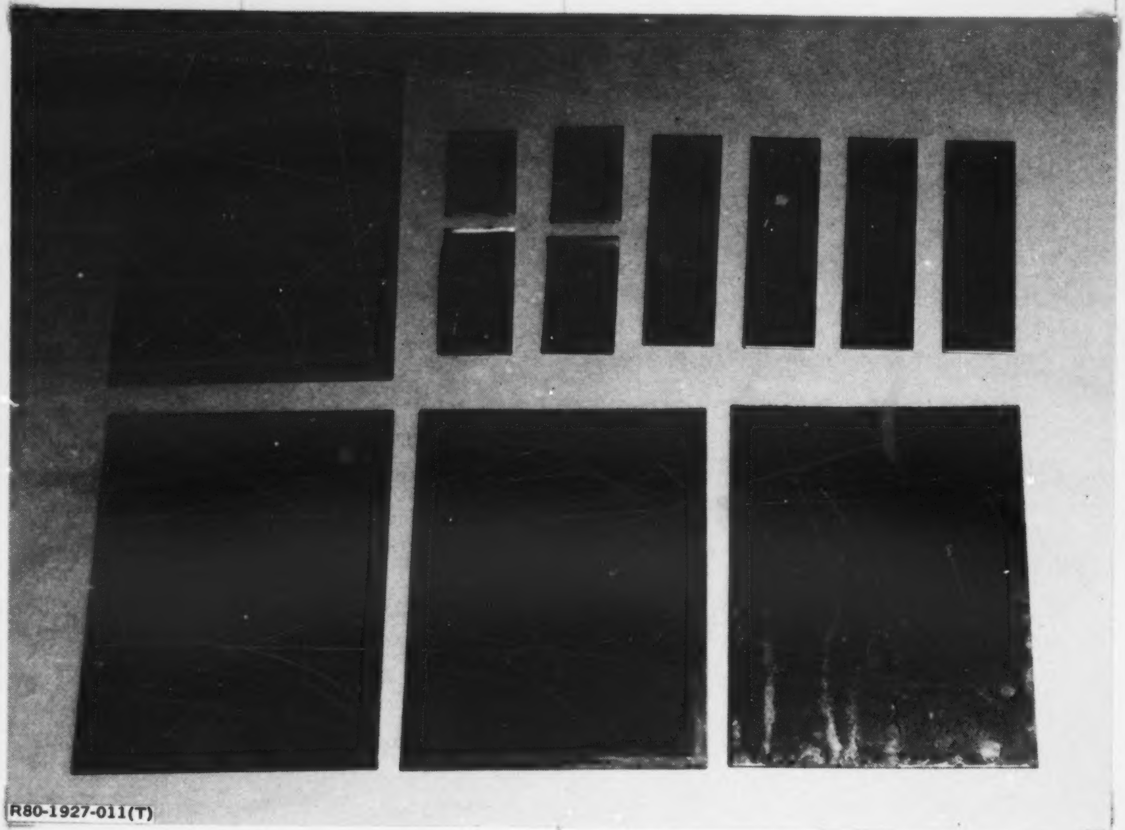

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Figure 6. Spray-and-Bake Aluminum - Hi Kote 3-Coated Test Panels.

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TABLE 6. ION-VAPOR-DEPOSITED (IVD) ALUMINUM - PRELIMINARY EVALUATION

Property	IVD Aluminum with Chromate Conversion Coating	Cyanide Cadmium with Chromate Conversion Coating
Appearance	Smooth	Smooth
Adhesion	Excellent	Good
Corrosion resistance, hr in 5% salt spray substrate - 4130	Pass 1000	Fail 576 (avg)
Thickness, μm (mil)	20.4 - 30.4 (0.8 - 1.2)	1.3 - 5.1 (0.05 - 0.2)

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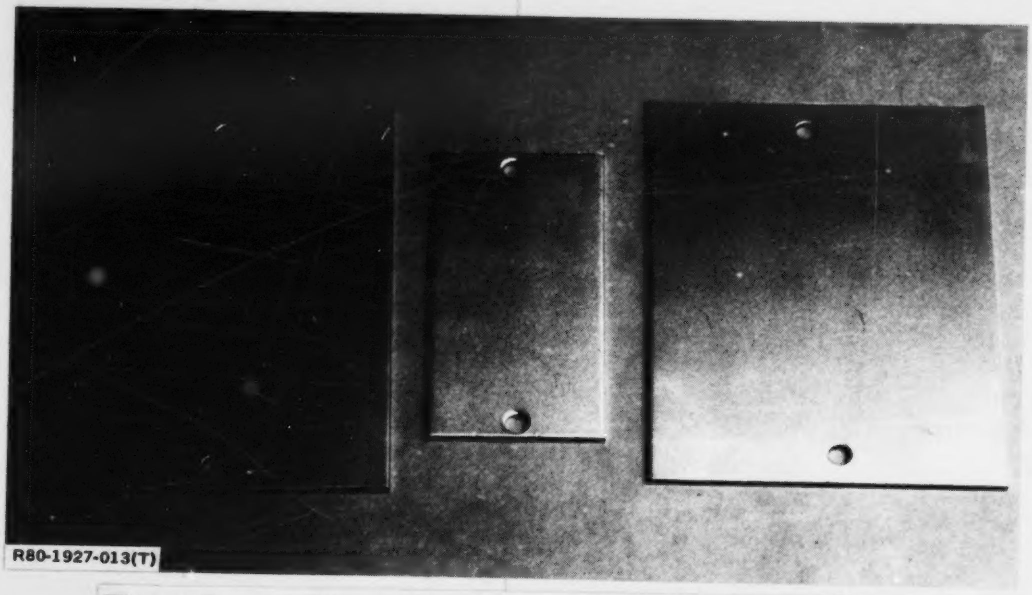

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Figure 7. Ion-Vapor-Deposited (IVD) Aluminum Test Panels.

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TABLE 7. CORROSION RESISTANCE OF CYANIDE CADMIUM REPLACEMENT COATINGS

		5% Salt Spray: Hours to failure							Empigard I applied to:		
				Mechanical Plating		Spray & Bake Aluminum			Mechanical Plating		Spray & Bake Aluminum
Substrate	Panel No.	Cyanide Cadmium	Non-Cyanide Cadmium	Cadmium	Tin-Cadmium	Alumazite Z	Hi Kote 3	IVD Aluminum	Cadmium	Tin-Cadmium	Hi Kote 3
	1	2616	1224	6192*	480				484	551	
4340 Steel	2	2208	2928	624	768				2796	771	
	3	2616		2616	456				2868	843	
	4	2616		624	528				2868		
	AVG	2514 Class 3, Type II	2076 Class 1, Type II	2514^{+} Class 2, Type II	558 Class 2, Type I				2254	722	
	1	1464		3912	504				771	168	
D6AC Steel	2	2016		6192*	456				2700	387	
	3	1656		2616	600				2700		
	4	3552		1368	504				2868		
	AVG	2172 Class 2, Type II		3522^{+} Class 1, Type II	516 Class 2, Type I				2259	277	
	1	672				670	312	1680			336
4130 Steel	2	432				730	168	1800			336
	3	1104					408	1800			
	4	96					312	1728			
	AVG	576 Class 1, Type II				700	300	1752			336

*Not tested to failure

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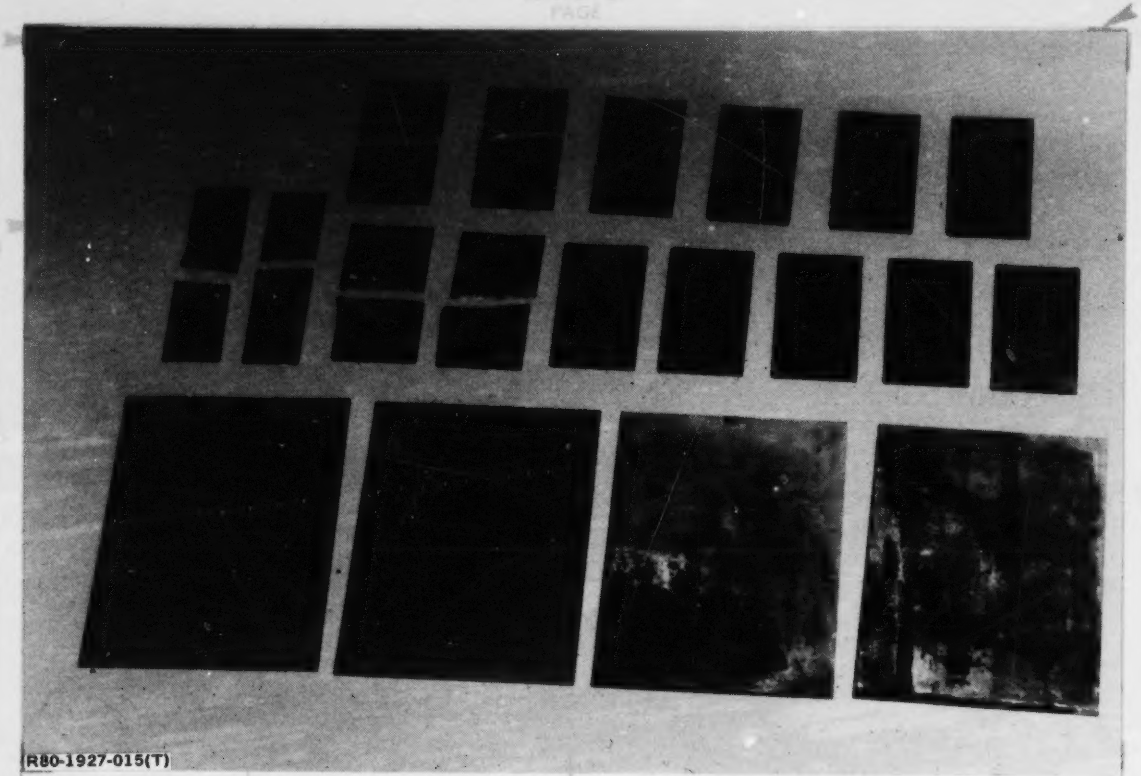

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Figure 8. Cyanide Cadmium-Plated Test Panels.

no effect on or slightly decreased the corrosion resistance of the other coatings evaluated. Empire Corporation suggests that this resulted from not applying the Empigard I coating immediately after the plating operation, a procedure which should be used to obtain improved corrosion resistance over the coated material without Empigard.

Hydrogen Embrittlement

Sustained load testing was conducted on selected coating systems to evaluate the potential for embrittlement failure resulting from hydrogen absorption. Notched tensile specimens (Fig. 9) made from 300M and 4340 high-strength steels were coated with the various coating systems and subjected to a static load at 75% of the ultimate notched tensile strength for 200 hr or until failure. The ultimate notched tensile strength was determined by continuously loading uncoated specimens until failure. The notch tensile strength of the 4340 steel was established at 56.5 MPa (389.3 ksi), and that of the 300M steel was 59.4 MPa (409.6 ksi) (Table 8). Each of the cyanide cadmium-plated controls exceeded 200 hr under sustained load. The cyanide cadmium specimens were plated to 13 μm (0.6 mil) and baked at 191 °C (375 °F) for 24 hr for embrittlement relief. Non-cyanide cadmium plating was evaluated under three conditions on 4340 steel. Three specimens, plated to 13 μm (0.5 mil) and baked at 191 °C (375 °F) for 3 hr, exceeded 200 hr under sustained load. Specimens plated to 18 μm (0.7 mil) required a 23-hr bake at 191 °C (375 °F) for complete embrittlement relief; of those baked for only 3 hr, one failed at 27 hr under sustained load. The remaining specimens were plated to 18 μm (0.7 mil), two of which were baked for 3 hr and the three baked for 23 hr; all exceeded 200 hr under sustained load.

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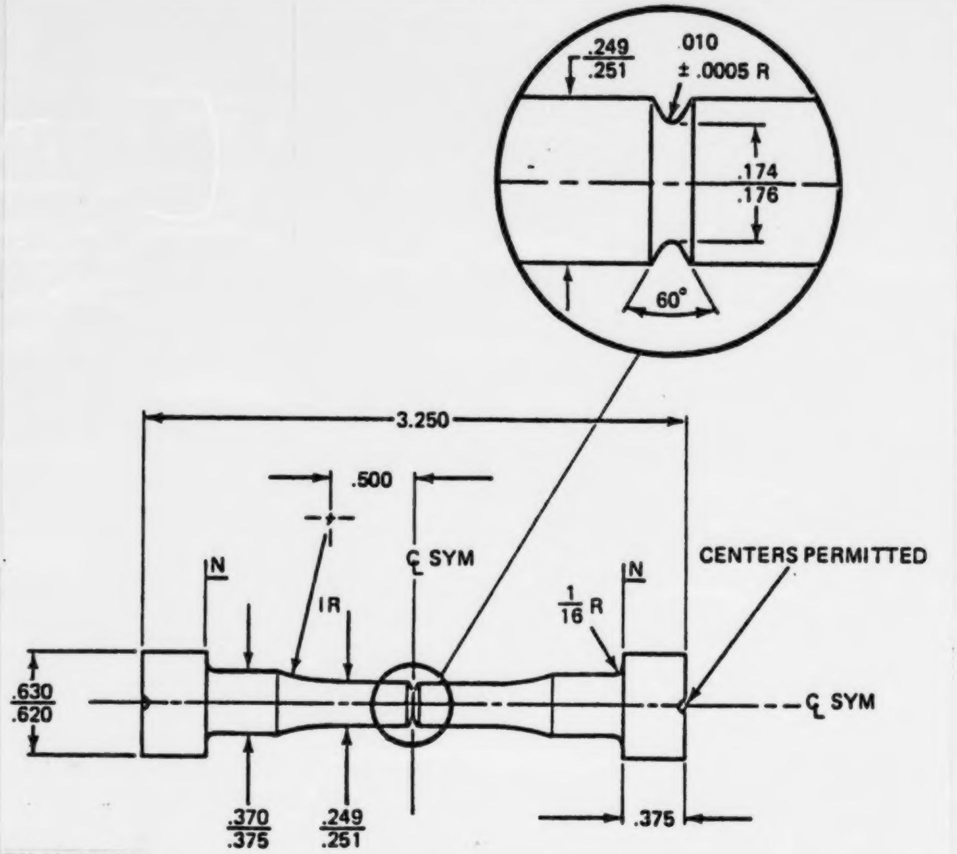
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Figure 9. Notch Tensile Strength Specimen Configuration ($K_T = 3.05$)

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TABLE 8. Notched-Tension Sustained-Load Tests - Cyanide Cadmium Replacement Coatings

Coating System	Sub-strate	Load (0.75 x ULT*), KN (LB)	Spec No.	Test Duration,** Hr		Remarks
			19	262	NF‡	
Cyanide cadmium plated (control)	4340	31.2(7020)	21	262	NF	13 μm (0.5 mil), baked 24 hr @ 191°C (375°F)
			27	262	NF	
	300M	32.8(7365)	2A	262	NF	13 μm (0.5 mil), baked 24 hr @ 191°C (375°F)
			3A	262	NF	
Non-cyanide cadmium plated			5	214	NF	
	4340	31.2(7020)	6	262	NF	13 μm (0.5 mil), baked 3 hr @ 191°C (375°F)
			31	213	NF	
			8	27	F‡	
	4340	31.2(7020)	12	214	NF	18 μm (0.7 mil), baked 3 hr @ 191°C (375°F)
			13	213	NF	
	4340	31.2(7020)	7	287	NF	
			26	287	NF	18 μm (0.7 mil), baked 23 hr @ 191°C (375°F)
			33	286	NF	
Mechanically plated cadmium			1	259	NF	
	4340	31.2(7020)	2	258	NF	13 μm (0.5 mil)
			3	200	NF	
	300M	32.8(7365)	4A	257	NF	13 μm (0.5 mil)
			5A	259	NF	
Alumazite Z Spray-and-bake			4	215	NF	
	4340	31.2(7020)	10	215	NF	5-13 μm (0.2 - 0.5 mil)
			23	216	NF	

*Ultimate load:

Substrate	Spec No.	Ult load kn(lb)	Notch Tensile Strength MPa(ksi)
	14	41.5(9330)	56.3(388.1)
4340	15	41.7(9365)	56.5(389.5)
	28	41.7(9380)	56.6(390.2)
	Avg	41.6(9358)	56.5(389.3)
300M	1A	43.7(9830)	59.4(409.6)

**Minimum requirement: 200 hr @ 75% of ultimate notch tensile strength

‡NF - no failure F- failure

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Mechanically plated cadmium was applied to 4340 and 300M steel specimens to 13 μm (0.5 mil). Each of these specimens exceeded 200 hr under sustained load. Alumanite Z spray-and-bake coated specimens were also subjected to the sustained load; the three specimens exceeded 200 hr with no failure.

Comparison of Selected Systems

The performance characteristics of each of the systems selected as replacements for cyanide cadmium plating approximate those of the cyanide cadmium control coating. Adhesion, corrosion resistance, and hydrogen embrittlement were evaluated for each of the selected coatings (Table 9). The adhesion of non-cyanide cadmium plate, spray-and-bake aluminum, and IVD aluminum was excellent. Satisfactory adhesion was obtained with the mechanical plating and cyanide cadmium control coatings. Each of the selected coatings, except the spray-and-bake aluminum, had a chromate conversion coating applied to improve corrosion resistance. Although the average corrosion resistance of the non-cyanide cadmium-plated 4340 steel is not as good as that of the cyanide cadmium plate (2076 hr compared to 2514 hr), it is very good compared to the minimum requirement for cadmium-plated steel (QQ-P-416, 168 hours, minimum). Mechanically plated cadmium showed equivalent or better corrosion resistance on both 4340 and D6AC steels compared to the cyanide cadmium plating. While the scatter in the data for most of these coatings is very large, indicating that the average is not a good measure of the overall corrosion resistance, each individual value is greater than the minimum requirement specified in QQ-P-416 and MIL-C-81562. Relative to these specifications, the corrosion resistance of the selected coatings can be considered to be excellent.

IVD aluminum showed excellent corrosion resistance on 4130 steel (1752 hr compared to 576 hr for cyanide cadmium) with minimal variation in results for the IVD aluminum. The Alumazite Z coating also showed slightly improved corrosion resistance over the cyanide cadmium plate for 4130 steel (700 hr compared to 576 hr). No hydrogen embrittlement occured as a result of coating application for any of the selected coatings.

Economic and Environmental Evaluation

The economic factors of the selected systems are related to the equipment requirements for each system, the cost of coating materials, and the steps required for environmental conformance for each system. Each of the systems selected as replacements for cyanide cadmium electroplating has advantages relative to both the cyanide systems and the other replacement systems (Table 10). The selection of a particular replacement system will depend on a number of factors defined by the specific application and circumstances.

The equipment requirements for non-cyanide cadmium are essentially the same as those for cyanide cadmium electroplating. Differences include tank construction material and anode type. Because these differences are minimal, the installation cost for new equipment would be approximately equal for cyanide and non-cyanide electroplating. Conversion costs would also be low for the two types of electroplating. Tanks must be lined and equipment coated with PVC or other corrosion-resistant material; no other equipment changes are required. Because no cyanides are present in this alternative cadmium coating system, the cyanide destruction step of the waste treatment process is eliminated. The chemicals required for solution makeup and maintenance are more costly for the Kadized non-cyanide system than those required for the Enthobrite cyanide cadmium system.

The advantages of mechanically plated cadmium include elimination of the cyanide destruction requirement, because no cyanides are present in the mechanical plating system, and elimination of the potential for hydrogen embrittlement. Because special equipment is required for mechanical plating, the conversion cost will not be low; however, for small parts such as fasteners, this system has excellent potential.

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TABLE 9. CYANIDE CADMIUM REPLACEMENT COATINGS - PERFORMANCE SUMMARY

System	Adhesion (bend test)	Corrosion resistance, hr to failure[**]			Hydrogen embrittlement
		4130	4340	D6AC	
Cyanide cadmium electroplate[*]	Good	576	2514	2172	Pass
Non-cyanide cadmium electroplate[*] (Kadizid)	Excellent	–	2076[‡]	–	Pass
Mechanical cadmium plating[*] (Transiflo)	Good	–	2514[‡]	3522	Pass
Spray-and-bake aluminum (Alumazite Z)	Excellent	700	–	–	Pass
IVD aluminum[*] (Ivadize)	Excellent	1752	–	–	–

[*] chromate conversion coating applied to plated panel
[**] average of four panels
[†] average of two panels

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Conversion to spray-and-bake aluminum coatings should be relatively inexpensive, providing that curing ovens and paint spray booths are available. Conventional paint spray equipment is used to apply spray-and-bake aluminum coatings. Because cyanide and cadmium are not used, waste treatment costs are minimal. An organic solvent recovery system is required for pollution control. If a water-borne spray-and-bake aluminum coating system is developed in the near future, a wide variety of applications will be available for these coatings because they can be formulated for maximum performance.

Special equipment is required for the application of the ion-vapor-deposited (IVD) aluminum coating. Equipment can be designed for coating large parts or for continuous coating of small parts such as fasteners. No waste treatment is required for the IVD process; the potential hazards of both cyanides and cadmium have been eliminated. Additional performance advantages are also available with the IVD-aluminum coating--no hydrogen embrittlement occurs in IVD-coated parts and the maximum use temperature is 510°C (950°F) compared to 232°C (450°F) for cadmium coatings.

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TABLE 10. CYANIDE CADMIUM REPLACEMENT COATINGS - ADVANTAGES AND DISADVANTAGES

Cyanide Cadmium	Non-Cyanide Cadmium	Mechanical Plating	Spray-and-Bake Aluminum	IVD Aluminum
		Advantages		
• Readily available	• Reduces waste treatment req	• Eliminates cyanides	• Eliminates cyanides	• Eliminates cyanides
• Techniques well-established	• Eliminates cyanides	• Eliminates hydrogen embrittlement	• Eliminates cadmium	• Eliminates cadmium
• Proven system	• Conversion costs low		• Reduces waste treatment req	• Eliminates waste treatment req
			• Eliminates hydrogen embrittlement	• Eliminates hydrogen embrittlement
			• Uses conventional spray equipment	• Use to 510°C (950° F) (CD limited to 232° C 450° F)
			• Can be formulated for max performance	
		Disadvantages		
• Uses toxic chemicals	• Higher makeup cost	• Requires new equipment	• Organic solvent collection system req	• Requires new equipment
• Requires costly waste treatment		• Part size limited		

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SECTION 5

REPLACEMENT COATINGS FOR ORGANIC SOLVENT PAINTS

APPROACH

This phase of the program was directed at evaluating potential replacement systems for organic solvent paints and maskants. The approach taken involved the evaluation of several water-borne paints, fluidized-bed powder coatings, electrostatic powder spray coatings, and water-borne maskants.

STUDY AREAS

Evaluation of replacement coatings for organic solvent paints and maskants involved the following:

- Selection of substrates, pretreatment procedures, and application techniques
- Preliminary testing and evaluation of coatings
- Final characterization of selected coatings
- Outdoor exposure of selected coatings
- Economic and environmental evaluation.

BACKGROUND

Paints are used in many decorative and corrosion protection applications for home appliances, automobiles, aluminum siding, garden furniture, aircraft skins, and many other applications. Maskants are used to protect parts from being etched during the chemical milling process. Currently used paint and maskant systems employ organic solvents as volatile components. Toxicity of these organic solvents requires that stringent pollution-control procedures be followed in the use of these systems to meet established standards for maximum discharge levels. Additional problems associated with using organic solvent paints include flammability, rising prices, and decreasing availability of the solvents. The use of water as the volatile component virtually eliminates these problems. Several of the water-borne paints currently available have been designed to withstand the applications mentioned above. Powder coatings, applied by fluidized bed or electrostatic powder spray, also eliminate the problems associated with organic solvents. Many different resins have been used for these 100% solids resin systems, each with a different set of properties. Formulations of the same basic resin can be varied to adjust some of these properties. Several water-borne maskants have been developed to replace the organic solvent-type systems currently used for chemical milling applications. The candidate systems evaluated as potential replacement coatings for organic solvent paints and maskants are itemized in Tables 11, 12, and 13.

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TABLE 11. WATER-BORNE PAINT SYSTEMS EVALUATED

Paint system	Manufacturer	Type	Cure	Primer[*]
Aquathane WA-1001	Sterling Lacquer	Epoxy - acrylic	Air dry	Aquathane WA-1017
Aquathane II	Sterling Lacquer	Urethane	Air dry	Aquathane WA-1017
Aqualure 481-W-02114	Glidden	Acrylic	300°F - 15 min	Aqualure 631-L-128 (acrylic)
Aqualure 634-W-804	Glidden	Polyester	350°F - 15 min	Aqualure 631-L-128 (acrylic)
SAF-T 300-W-39	U.S. Paint, Lacquer, & Chemical	Epoxy	Air dry	SAF-T-300-G-19A
SAF-T 300-W-44	U.S. Paint, Lacquer, & Chemical	Urethane	Air dry	SAF-T 300-G-19A
Carboset 514H/Epon 828 Formulation	BF Goodrich Resin/Dow Resin	Epoxy - acrylic	300°F - 15 min	None required
WB 304	Bee Chemical		300°F - 20 min	None required
2030-119	De Soto, Inc	Acrylic	Air dry	2030-118
LP-3724	Union Carbide	Styrene-acrylic	350°F - 10 min	LP-3778
G-9-1 A	Rohm & Haas	Acrylic	Air dry	PO-9-16[‡]
G-9-1 B	Rohm & Haas	Acrylic	Air dry	PO-9-16[‡]

[*] Epoxy primer MIL-P-23377 also evaluated with some of these systems

[‡] PO-9-16 selected as best of six primers evaluated from Rohm & Haas

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TABLE 12. POWDER COATINGS EVALUATED

Powder	Source	Application technique	Applied by	Cure	Substrate	Primer
Acrylic (154-W-04110)	Glidden	Electrostatic spray	Interrad	340°F – 30 min 375°F – 20 min 375°F – 20 min	Aluminum Aluminum Steel	
Epoxy (PLAS 5027)	Plastonics	Electrostatic spray	Plastonics	400°F – 10 min 400°F – 10 min	Aluminum Steel	
Polyamide (Grilamid)	Emser industries	Electrostatic spray	Interrad	420°F – 6 min 420°F – 16 min	Aluminum Steel	HV-BO294 HV-BO294
Polyester (PLAS 4009)	Plastonics	Electrostatic spray	Plastonics	400°F – 10 min 400°F – 10 min	Aluminum Steel	
Polyester (155-W-04130)	Glidden	Electrostatic spray	Interrad	340°F – 30 min 375°F – 20 min 375°F – 20 min	Aluminum Aluminum Steel	
Polyester (156-W-04102)	Glidden	Electrostatic spray	Interrad	340°F – 30 min 375°F – 20 min 375°F – 20 min	Aluminum Aluminum Steel	
Nylon (PLAS 1008)	Plastonics	Electrostatic spray	Plastonics	425°F – 10 min 425°F – 10 min	Aluminum Steel	Nylon primer Nylon primer
Nylon 11	LNP, Rilsan	Fluidized bed	Grumman	500°F – 1 to 3 min 500°F – 1 to 3 min	Aluminum Steel	Primer P (Rilsan)
Vinyl	Interrad	Electrostatic spray	Interrad		Aluminum	

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TABLE 13. WATER-BORNE MASKANTS EVALUATED

Maskant	Manufacturer	Application	Cure
A 14-61-1 primer A 14-47-1 mask	Adcoat	Primer dip & dry 25 min Mask dip & dry 25 min	Pass under infra-red heater
Dapcoat 10200	D Aircraft Products	Sensitizer dip & hold 30 sec Mask dip & hold 2-3 min	73°F/1/2 - 1 hr 75 - 110°F/4-24 hr + 200°F/1 hr
Turcoform Mask 700	Turco	Spray (3 coats)	73°F/24 hr or 73°F/6 hr + 220°F/1 hr

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SUBSTRATE PRETREATMENT AND COATING APPLICATION

Typical substrates that are protected with paint include 2024-T3 aluminum and 1010-1020 steel. Pretreatment of the aluminum includes solvent clean, alkaline clean, de-oxidize, and alodine or anodize. The steel is pretreated by vapor degreasing, alkaline cleaning, and vapor blasing. Primer was used on both aluminum and steel where required.

The water-borne paints were prepared according to manufacturers' recommended procedure (Table 14), and applied with a DeVilbiss Type EGA, Series 502 spray gun at 2.0 to 2.7 atm (30 to 40 psi) in a laboratory spray booth. One to three components were mixed and reduced with water to spraying viscosity (18 to 23 sec in No. 2 Zahn Viscometer) before spraying. The coatings were either bake-cured or air-dried for 7 days before testing.

Three of the electrostatic powder spray coatings were applied by Plastonics, Inc., Hartford, Conn. The panels, pretreated as described above, were cleaned with Detrex vapor degrease and coated with epoxy and polyester by spraying for 2 to 3 sec per panel. The coatings were then cured for 10 min at 204°C (400°F) (Ref. Table 12). Preparation for the nylon coating included pretreatment as described above, cleaning in a three-stage iron phosphate wash, and priming. Nylon was sprayed on for 2 to 3 sec per panel and cured for 10 min at 218°C (425°F). The remaining electrostatic powder spray coatings were applied by Interrad Corp., Stamford, Conn. These panels were pretreated as described above and coated with polyester (two types), acrylic, and polyamide by spraying 2 to 3 sec per panel. Two cure cycles were evaluated for the acrylic and polyesters: 30 min at 171°C (340°F) and 20 min at 191°C (375°F). The polyamide was cured for 6 to 16 min at 216°C (420°F). A primer was utilized for the polyamide powder.

One topcoat, Carboset 514 H/Epon 828, was prepared from a resin base without pigment for simplicity according to a formulation suggested by BF Goodrich (Ref. 2). Although this topcoat failed the impact resistance test, it stood up well in the other tests. This coating was prepared to show that water-borne paint systems other than those currently on the market can be formulated to provide the desired properties. Modifications to this formulation, not within the scope of this study, can be evaluated to establish the optimum combination of properties for a specific application.

Application of the powder coatings by the fluidized bed technique was done using a laboratory unit. Air to the unit was preheated in a Chromalox preheater to dry the powder prior to coating. Two beds, 30.5 cm (1.0 ft) in diameter and 0.762 m (2.5 ft) high, with ceramic diffuser plates were used.

TABLE 14. WATER-BORNE PAINTS - APPLICATION PARAMETERS

Paint System	Components	Mixing Ratio	Thinning Solvents	Cure
Polyurethane topcoat Mil-C-81773; Solvent borne system (control)	Component I Component II	3 vol 1 vol	Organic Solvents	Air-dry 7 days
Aquathane WA-1017 primer	Primer base WA1017 Catalyst WA1018	1 vol 3 vol	Water	Air-dry 2-4 hr; Apply topcoat
Aquathane WA-1001 topcoat	Base Catalyst	3 vol 1 vol	Water	Air-dry 5 days
Aquathane II topcoat	Component I Component II Component III	40 vol 7 vol 1 vol	Water	Air-dry 8-10 hr
Aqualure 631-L-128 primer	Premixed	–	Water	15 min @ 107°C (225°F); Apply topcoat
Aqualure 634-W-804 topcoat	Premixed	–	Water	15 min @ 177°C (350°F)
Aqualure 481-W-02114 topcoat	Premixed	–	Water	15 min @ 138-149°C (280-300°F)
Saf-T-300-G-19A primer	Premixed	–	Water	Air-dry 1-2 hr Apply topcoat
Saf-T-300-W-39 topcoat	300-W-39A 300-W-39B	6 vol 1 vol	Water	Air-dry overnight
Saf-T-300-W-44 topcoat	Premixed	–	Water	Air-dry overnight
Carboset 514H/Epon 828 formulation topcoat	Carboset 514H Demineralized water Epon 828 resin	675 wt 225 wt 60 wt	Water	15 min @ 149°C (300°F)
WB 304 topcoat	Premixed	–	Water	20 min @ 149°C (300°F)
2030-118 primer	2030-118 2030-132	12 vol 1 vol	Water	Air-dry 1-4 hr; Apply topcoat
2030-119 topcoat	2030-119 2030-132	10 vol 1 vol	Water	Air-dry 7 days

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TABLE 14. (continued)

Paint System	Components	Mixing Ratio	Thinning Solvents	Cure
LP-3778 primer	Premixed	–	Water	20 min @ 177°C (350°F); apply topcoat
LP-3724 topcoat	Premixed	–	Water	10 min @ 177°C (350°F)
PO-9-16 primer	Premixed	–	Water	Air-dry 1-4 hr; Apply topcoat
G-9-1 A	Premixed	–	Water	Air-dry 7 days
G-9-1 B	Premixed	–	Water	Air-dry 7 days
Epoxy primer MIL-P-23377 Solvent borne system (control)	Component I Component II	1 vol 1 vol	Organic Solvents	Air-dry 1 hr; Apply topcoat

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The beds were filled about one-half with powder and then fluidized to about two-thirds to a uniform light bubbling action. Application of the powder required that the part be preheated, dipped in the powder, and post-cured. The optimum temperature for preheat and postcure, and the time for postcure, are dependent on the mass of the part and the type of resin used.

Coating conditions were established for application of nylon on both steel and aluminum by evaluating the thickness and appearance of panels coated under various preheat temperatures, bake times and temperatures, and number of coats. Two sources of nylon 11 resin powder (LNP and Rilsan) were tested. Although slight differences in bake time and coating thickness were apparent in the powders from the two sources, these differences were not considered significant. The following conditions were found to provide uniform, continuous coatings without discoloration:

- Single dip on steel:
  Temperature - 260°C (500°)
  Bake time - 1 to 1.5 min
  Panel size - 0.09 x 7.6 x 15.2 cm
  (0.035 x 3 x 6 in.)

- Two dips on steel:
  Temperature - 260°C (500°F)
  Bake time - 1st dip - 1 to 2 min
  2nd dip - 1 to 2 min
  Panel size - 0.09 x 7.6 x 15.2 cm
  (0.035 x 3 x 6 in.)

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- Two dips on aluminum:
  Temperature - 260°C (500°F)
  Bake time - 1st dip - 1 to 2 min
  2nd dip - 1 to 1.5 min
  Panel size - 0.05 x 7.6 x 15.2 cm
  (0.020 x 3 x 6 in.)

- Three dips on aluminum:
  Temperature - 260°C (500°F)
  Bake time - 1st dip - 0.5 to 1.5 min
  2nd dip - 0.5 to 1.5 min
  3rd dip - 0.5 to 1.5 min
  Panel size - 0.05 x 7.6 x 15.2 cm
  (0.020 x 3 x 6 in.)

The candidate water-borne maskants were applied to aluminum panels by the manufacturers according to their standard procedures (Ref. Table 13).

TESTING AND EVALUATION OF ORGANIC-SOLVENT PAINT REPLACEMENT COATINGS

Testing the potential replacement coatings for organic solvent paints and maskants, i.e., water-borne paints, powder coatings, and water-borne maskants, was performed to determine the best system available for each type and to establish the relative merits of these types of coatings.

Water-Borne Paints

Twelve water-borne paint systems, obtained from eight companies, were evaluated (Ref. Table 11). Seven of these systems cure at room temperature; five require a higher temperature cure. Each of the systems, as well as the solvent-based control, were applied to aluminum panels that had been pretreated and alodined. Each topcoat was applied to the primer supplied for the system; in addition, several of the topcoats were applied to the solvent-based epoxy primer. The panels were then subjected to preliminary screening tests (Table 15). These tests were performed in accordance with the requirements of MIL-P-23377 and MIL-C-81773. Of the properties tested, adhesion, impact, and flexibility were considered most important. Test results (Fig. 10 through 13) were used to select the best coatings for further testing. Three of the coatings, Aquathane II with epoxy primer (air dry), Aqualure 634-W-804 with Aqualure primer (bake cure) and LP-3724 with LP-3779 primer (bake cure) provided the best combination of properties based on these criteria.

The surface appearance of the candidate coatings was judged to be satisfactory for each of the primers and topcoats. The films were all basically free of grit, seeds, craters, and blisters, although some of the films showed a slight mottling or orange-peel effect. The topcoat gloss as measured by the meter shown in Fig. 14, varied from 29 to 100 for the 60-degree geometry. The reading of 100 was obtained for the unpigmented Carboset 514H/Epon 828 formulation. A lower reading would be obtained if this formula were pigmented.

The coating adhesion and water resistance were determined in various tests, including primer adhesion (tape test after 24-hr water exposure), primer water resistance (visual and thumbnail after 18-hr water exposure), and topcoat anchorage (tape test after 24-hr water exposure). The primer adhesion and water resistance tests gave good results for all of the primers except Saf-T-300G-19A, which exhibited film softening and loss of adhesion in the tape test following water exposure. The two topcoats that were applied to the Saf-T-300G-19A primer (Saf-T-300W-39 and Saf-T-300-44) lifted from the primer in the tape test after water exposure (topcoat anchorage test). With Saf-T-300W-39, the primer also lifted from the substrate. Saf-T-300W-44 topcoat, which showed good results in the other screening tests, was applied to a solvent-based epoxy primer (meeting the requirements of MIL-C-23377) in

TABLE 15. WATER-BORNE PAINTS - SCREENING TEST RESULTS

Coating System	Primer Surface Appearance	Primer Adhesion	Primer Water Resistance	Topcoat Surface Appearance	Gloss (60°)	Topcoat Anchorage (Wet Tape)	Topcoat Flexibility (Conical Mandrel)	Topcoat Impact Resistance	Topcoat Heat Resistance	Topcoat Lube Oil Resistance
Control System										
Epoxy primer, Polyurethane topcoat	Satisfactory	Good	Slight discoloration; no other defect	Very slight mottling	89	Good	Pass	Pass 5.4 j (48 in.-lb)	No film defects; V Sl discoloration	No film defects; Slight discoloration
Air Dry Systems										
Aquathane WA-1017 primer WA-1017 topcoat	Satisfactory	Good	Discoloration; no other defect	Good	73	Good	Fail—film cracking at 0.5 in. dia.	Fail @ 2.3 j (20 in.-lb)	No film defects; Sl discoloration	No film defects; Slight discoloration
Aquathane WA-1001 primer Aquathane II topcoat	Satisfactory	Good	Discoloration ; no other defect	Very slight mottling	58	Poor adhesion, blistering	Pass	Pass 5.4 j (48 in.-lb)	No film defects; discoloration	No film defects; heavy discoloration
Epoxy primer Aquathane II topcoat	Satisfactory	Good	Slight discoloration; no other defect	Slight mottling	54	Good	Pass	Pass 5.4 j (48 in.-lb)	No film defects; discoloration	No film defects; discoloration
SAF-T-300G-19A primer SAF-T-300W-39 topcoat	Satisfactory	Fail—primer lifted from substrate	Fail—film softened	Mottling	68	Fail—topcoat & primer lifted from substrate	Fail—film cracking at 0.15 in. dia.	Fail @ 2.3 j (20 in.-lb)	No film defects; discoloration	No film defects; some discoloration

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TABLE 15. (continued)

Coating System	Primer Surface Appearance	Primer Adhesion	Primer Water Resistance	Topcoat Surface Appearance	Gloss (60°)	Topcoat Anchorage (Wet Tape)	Topcoat Flexibility (Conical Mandrel)	Topcoat Impact Resistance	Topcoat Heat Resistance	Topcoat Lube Oil Resistance
SAF-T-300G-19A primer SAF-T-300W-44 topcoat	Satisfactory	Fail—primer lifted from substrate	Fail—film softened	Good	55	Fail—topcoat lifted from primer	Pass	Pass 5.4 j (48 in.-lb)	No film defects; Sl discoloration	Film softened; discoloration
Formulated topcoat w/ Carboset 514H & Epon 828				Orange peel effect; surface not level	100	Good	Pass	Fail @ 2.3 j (20 in.-lb)	No film defects	Slight film softening (tacky); Slight discoloration
2030-118 primer 2030-119 topcoat	Satisfactory	Good	No effect	Good, Sl seeding	89	Very slight blistering	Fail—film cracking at 1.25 in. dia.	Fail @ 2.3 j (20 in.-lb)	No film defects; discoloration	Film softened; discoloration
PO-9-16 primer G-9-1A topcoat	Satisfactory	Good	Very slight discoloration	Sl mottling & seeding	43	Blistering; topcoat & primer spot lifted	Pass	Pass 6.8 j (60 in.-lb)		
PO-9-16 primer G-9-1B topcoat	Satisfactory	Good	Very slight discoloration	Good, Sl seeding	41	Some blistering, topcoat & primer spot lifted	Pass	Pass 6.8 j (60 in.-lb)	Some discoloration	Slight film softening; discoloration

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TABLE 15. (continued)

Coating System	Primer Surface Appearance	Primer Adhesion	Primer Water Resistance	Topcoat Surface Appearance	Gloss (60°)	Topcoat Anchorage (Wet Tape)	Topcoat Flexibility (Conical Mandrel)	Topcoat Impact Resistance	Topcoat Heat Resistance	Topcoat Lube Oil Resistance
Bake Cure Systems										
Aqualure 631-L-128 primer 481-W-02114 topcoat	Satisfactory (orange peel effect)	Good	Very slight film softening	Orange peel effect-surface not level	29	Good	Fail—film cracking at 0.4 in. dia.	Fail @ 2.3 j (20 in.-lb)	No film defects; Very slight discoloration	Slight film softening; Slight discoloration
Aqualure 631-L-128 primer 634-W-804 topcoat	Satisfactory (orange peel effect)	Good	Very slight film softening	Good	88	Good	Pass	Pass 5.4 j (48 in.-lb)	No film defects; very slight discoloration	Film softening; Slight discoloration
WB304 topcoat (no primer)	–	–	–	Thin coat, uniform seeding	7	Good	Pass	Fail @ 2.3 j (20 in.-lb)	Very slight discoloration	Film softened; discoloration
LP-3778 primer LP-3724 topcoat	Satisfactory	Very slight spot lifting	No effect	Good, very slight seeding & orange peel	69	Slight lifting along scribes	Pass	Pass 4.5 j (40 in.-lb)	No effect	Slight film softening; discoloration

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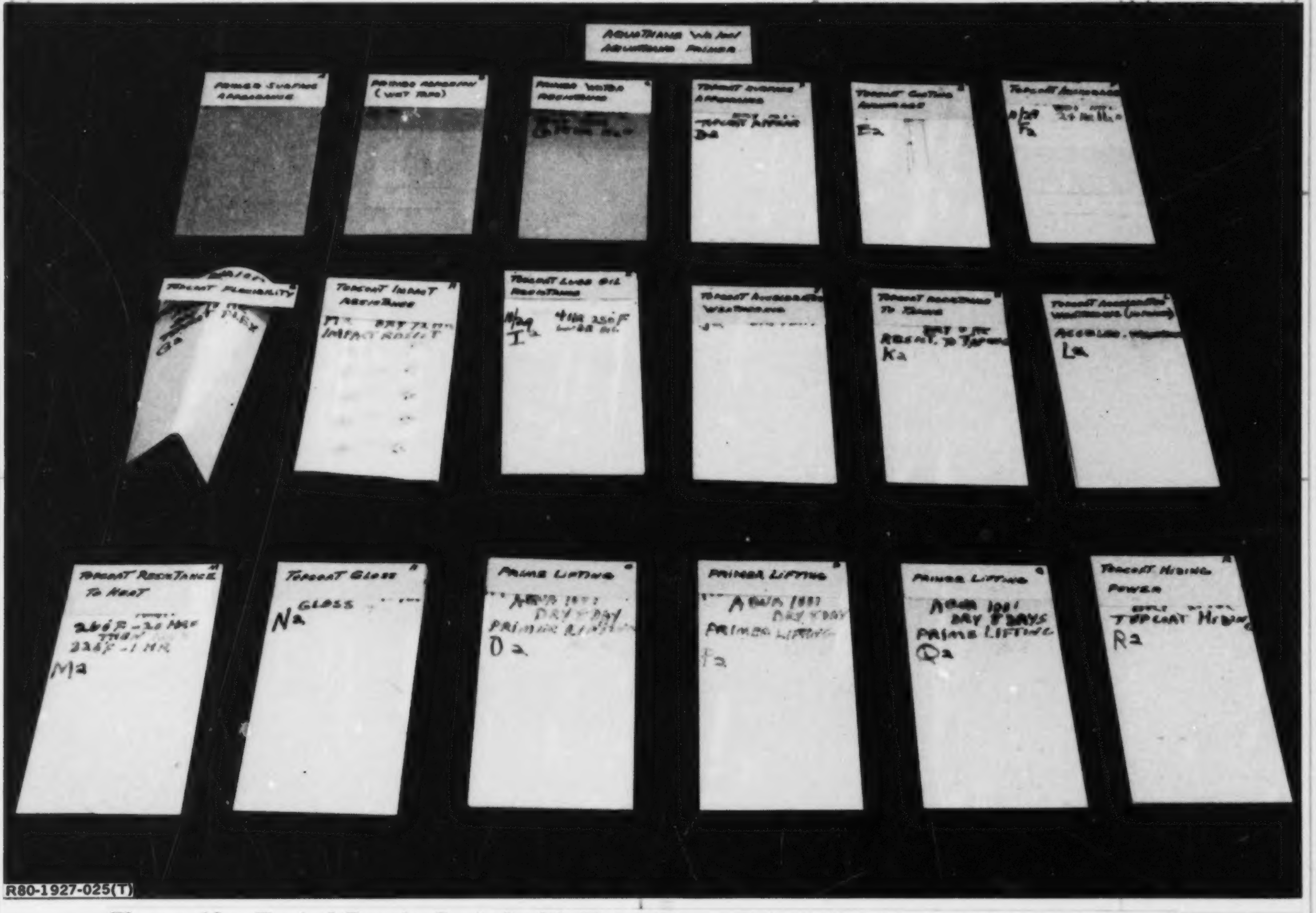
AQUATHANE WA 1001
AQUATHANE PRIMER
PRIMER SURFACE APPEARANCE
PRIMER ADHESION (WET TAPE)
PRIMER WATER RESISTANCE
TOPCOAT FLEXIBILITY
TOPCOAT IMPACT RESISTANCE
TOPCOAT LUBE OIL RESISTANCE
TOPCOAT RESISTANCE TO HEAT
TOPCOAT GLOSS
PRIMER LIFTING
TOPCOAT HIDING POWER
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Figure 10. Tested Panels Coated with Water-Borne Paint (Aquathane Primer/Aquathane WA-1001 Topcoat) - Screening Tests.

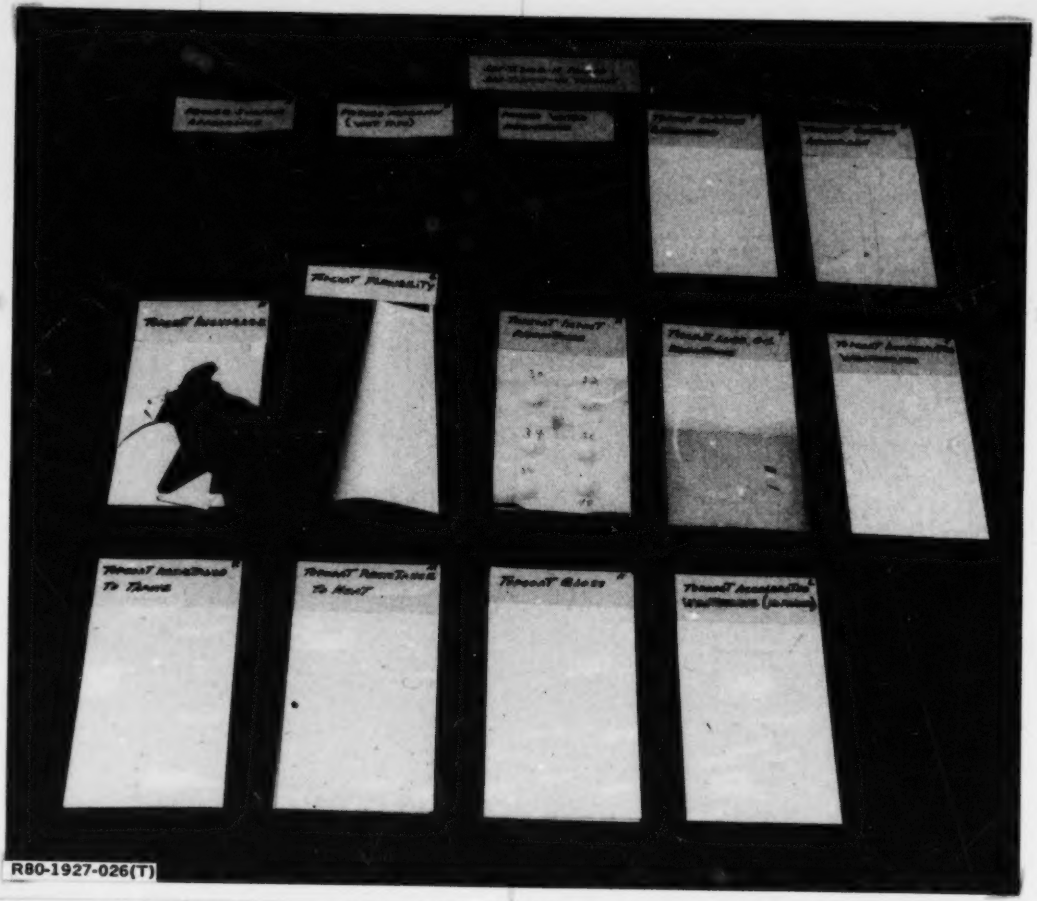
TOPCOAT FLEXIBILITY
TOPCOAT RESISTANCE TO HEAT
TOPCOAT GLOSS
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Figure 11. Tested Panels Coated with Water-Borne Paint (SAF-T-300G-19A Primer/SAF-T-300W-46 Topcoat) - Screening Tests.

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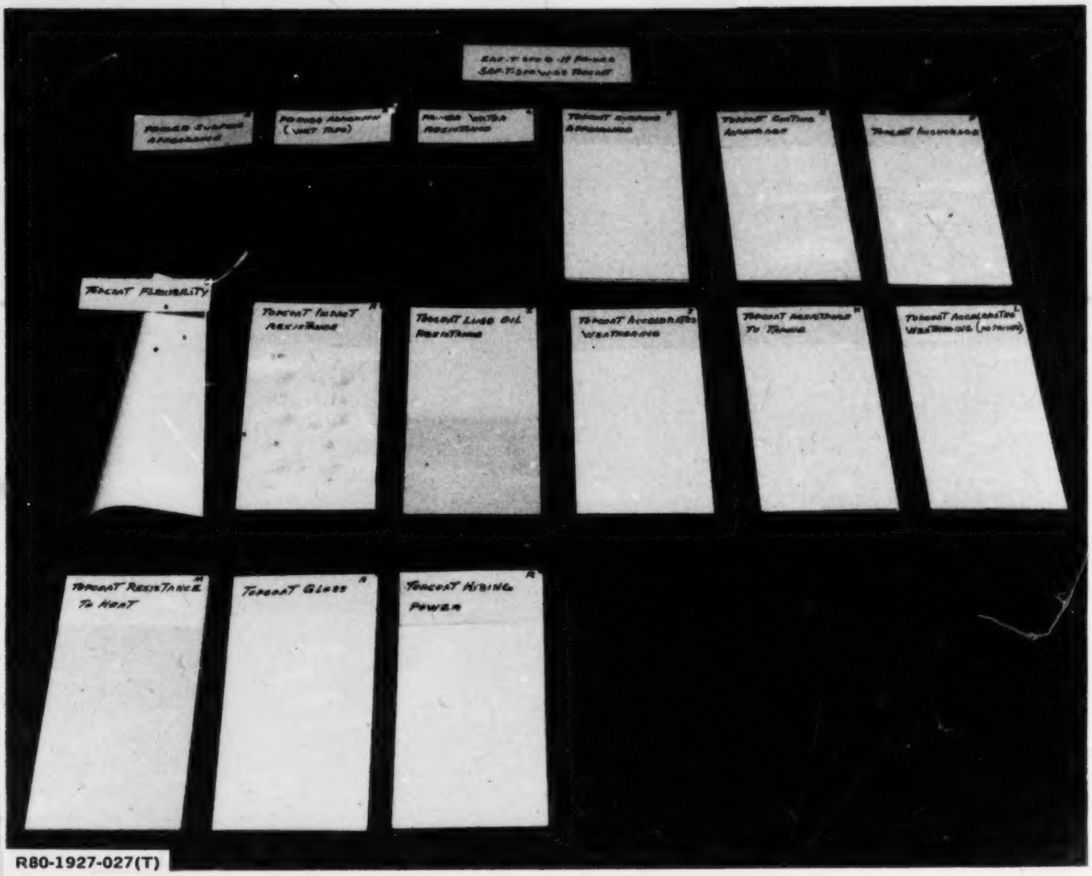
TOPCOAT FLEXIBILITY
TOPCOAT IMPACT RESISTANCE
TOPCOAT LUBE OIL RESISTANCE
TOPCOAT ACCELERATED WEATHERING
TOPCOAT ACCELERATED WEATHERING (NO PRIMER)
TOPCOAT RESISTANCE TO HEAT
TOPCOAT GLOSS
TOPCOAT HIDING POWER

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Figure 12. Tested Panels Coated with Water-Borne Paint (SAF-T-300G-19A Primer/SAF-T-300W-39 Topcoat) - Screening Tests.

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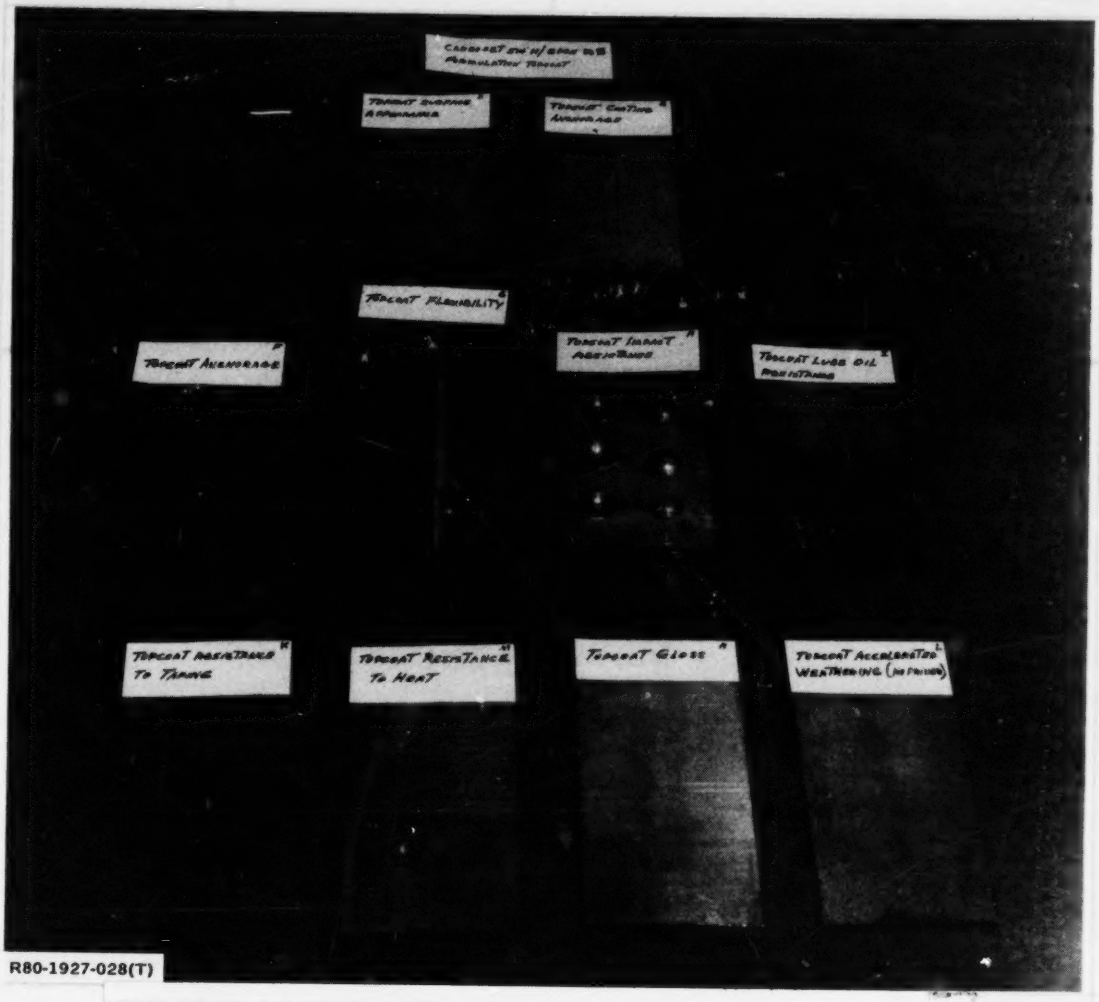
CARBOSET 514 H/EPON 828 FORMULATION TOPCOAT
TOPCOAT SURFACE APPEARANCE
TOPCOAT COATING ADHERENCE
TOPCOAT FLEXIBILITY
TOPCOAT ADHERENCE
TOPCOAT IMPACT RESISTANCE
TOPCOAT LUBE OIL RESISTANCE
TOPCOAT RESISTANCE TO TAPING
TOPCOAT RESISTANCE TO HEAT
TOPCOAT GLOSS
TOPCOAT ACCELERATED WEATHERING (no primer)

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Figure 13. Tested Panels Coated With Water-Borne Paint (Carboset 514H/Epon 828 Topcoat) - Screening Tests.

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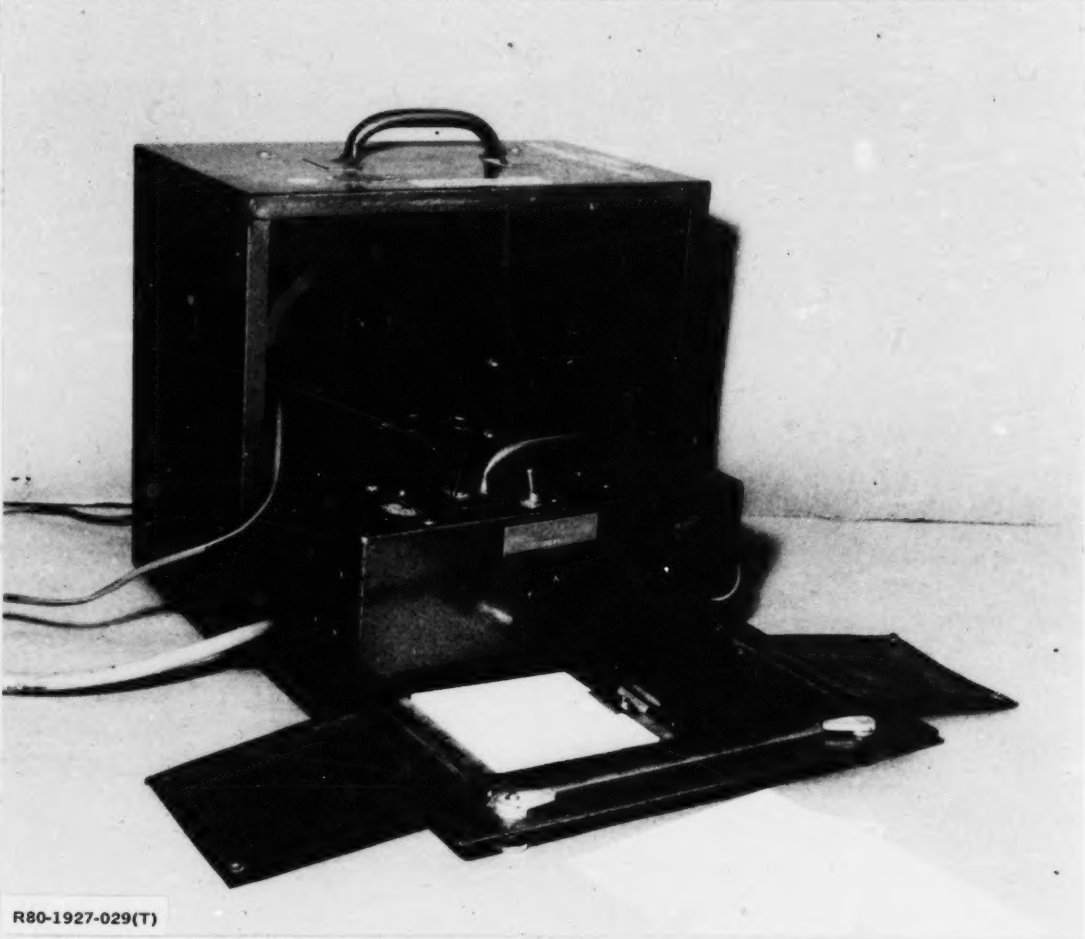

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Figure 14. Gardner 60° Gloss Meter - Model 2075

an effort to improve the adhesion. The topcoat adhesion improved somewhat with the epoxy primer, but remained unsatisfactory. Although the Aqualure 631-L-128 primer showed very slight film softening after water exposure, the primer adhesion and topcoat anchorage was good for both topcoats tested with that primer (Aqualure 481-W-02114 and Aqualure 634-W-804). Aquathane II topcoat with Aquathane WA-1017 primer showed poor adhesion after water exposure, with blistering of the topcoat and removal of the blistered topcoat from the primer in the tape test. Because the topcoat performed well in other screening tests, the solvent-based epoxy primer was used with this topcoat in an effort to improve the adhesion. This primer-topcoat combination provided good adhesion in the tape test after water exposure.

Although PO-9-16 primer showed good adhesion and water resistance, both the G-1-9A and the G-1-9B topcoats blistered following water exposure; the blistered spots lifted in the tape test. The remaining systems provided good primer and topcoat adhesion.

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The flexibility of the coatings was determined by bending painted panels over a conical mandrel (Fig. 15) having a minimum diameter of 0.32 cm (0.125 in.) and a maximum diameter of 5.1 cm (20 in.). No cracking, peeling, or loss of adhesion was observed at the smallest mandrel diameter for nine of the coatings tested. The Saf-T-300W-39 topcoat showed slight cracking at a mandrel diameter of 0.38 cm (0.15 in.). The Aqualure 481-W-02114 and Aquathane WA-1001 topcoats cracked at greater than 1.0 cm (0.4 in.) diameter. Film cracking of the 2030-119 topcoat occurred at 3.2 cm (1.25 in.) diameter. Each of the other coatings tested showed excellent flexibility, with no film cracking down to 0.32 cm (0.125 in.) diameter.

Each of the coatings that showed poor flexibility also performed poorly in the impact resistance test. The Carboset 514H/Epon 828 topcoat also had low impact resistance. The coatings were tested in a drop-ball impact tester (Fig. 16). The 0.91 Kg (2 lb) impacter is dropped from various heights above the panel up to 76.2 cm (30 in.), corresponding to 6.8 j (60 in.-lb) of energy absorbed by the coating under test, and the impacted area is examined for cracking and chipping. The Aquathane WA-1001, Aqualure 481-W-02114, Saf-T-300W-39, WB-304, and 2030-119 coatings each failed due to cracking at 2.3 j (20 in.-lb) or less in both direct and reverse impact.

The typical cracking pattern in the convex (reverse impact) case is a combination of concentric rings near the center of impact and radial cracks from the center. The cracking pattern observed in the concave (direct impact) case consists of large concentric rings near the outer edge of the impact area (Fig. 17).

The resistance of the topcoats to heat and lubricating oil was also determined in screening tests. Coated panels were subjected to 20 hr at 127°C (260°F) followed by 1 hr at 163°C (325°F), and visually examined to determine the effect of heat on the topcoats. The heat resulted in varying degrees of discoloration. No other film defects were observed as a result of the heat for any of the topcoats. Following 4 hr in diester lubricating oil at 121°C (250°F), panels were examined for blistering, film softening, and other film defects. The Saf-T-300W-44, 2030-119, Aqualure 634-W-804, and WB304 paint films softened after lubricating oil exposure. Slight softening of the Aqualure 481-W-02114, LP-3724, G-9-1B, and Carboset 514H/Expon 828 formulation paint films also resulted from lubricating oil exposure. The other paint films, including Aquathane WA-1001, Aquathane II, Saf-T-300W-39, and the epoxy-polyurethane control showed only varying degrees of discoloration as a result of lubricating oil exposure.

Based on the results of these preliminary screening tests, three coating systems were selected for further evaluation. Because air drying and oven curing systems have various advantages in cost and physical properties, one of each type was selected. Aquathane II with solvent-based epoxy primer was selected from the air-drying systems because of its superior adhesion, flexibility, and impact resistance. For the same reasons, Aqualure 634-W-804 with Aqualure primer and LP-3727 with LP-3778 primer were selected from the oven-curing systems. These three systems and the selected powder coating systems were subjected to final characterization testing (see the following section).

Powder Coatings

Eight powder coatings (acrylic, epoxy, polyamide, polyester (three types), nylon, and vinyl) were evaluated. Each of these resins was applied by electrostatic powder spray to steel and aluminum panels. Nylon was also applied to aluminum and steel panels by a fluidized bed. A primer was used for the nylon and polyamide coatings. Coated panels were subjected to preliminary screening tests (Tables 16 and 17) according to MIL-C-81773 procedures. As was the case with the water-borne paints, adhesion, impact, and flexibility were considered the most important of the properties tested.

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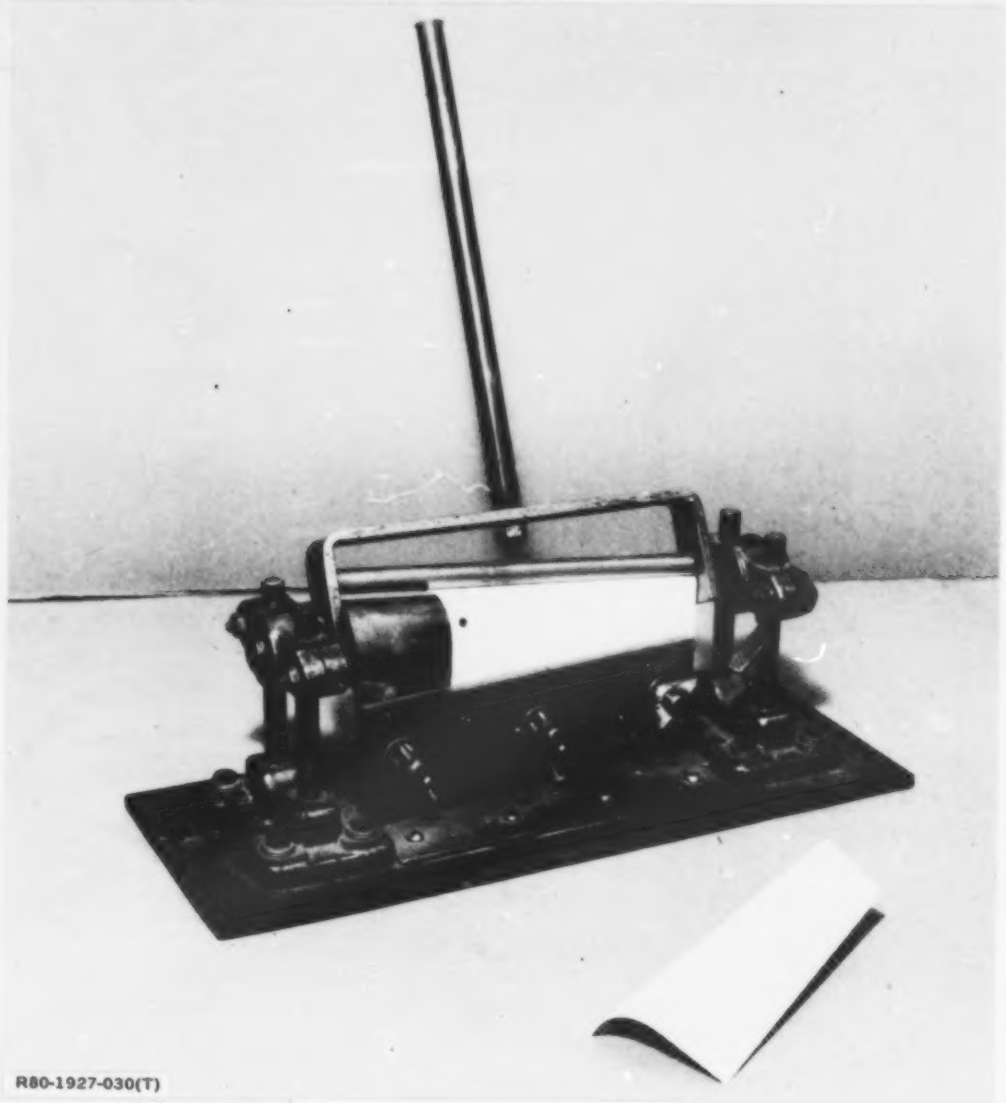

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Figure 15. Flexibility Tester.

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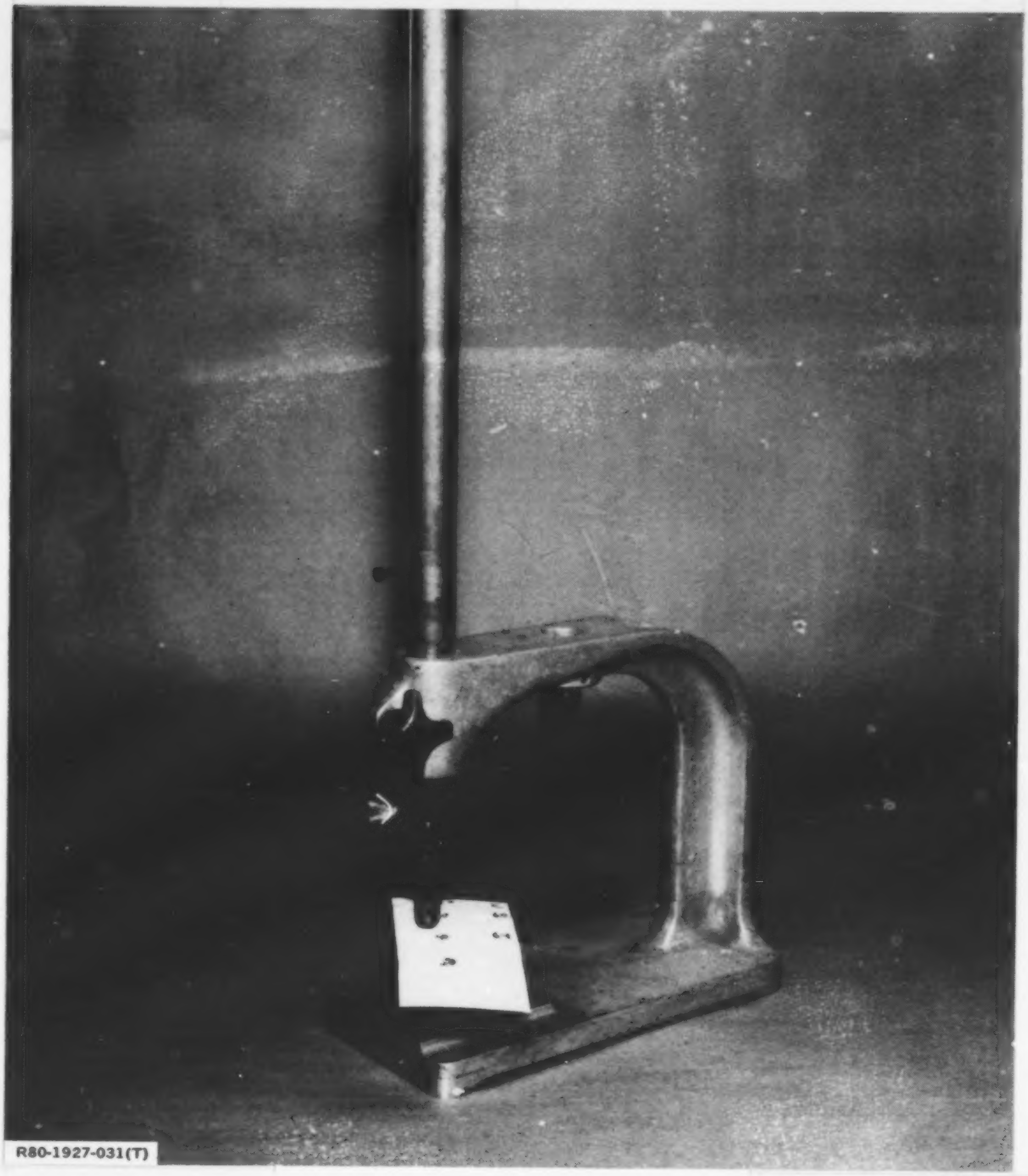

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Figure 16. Drop-Ball Impact Tester.

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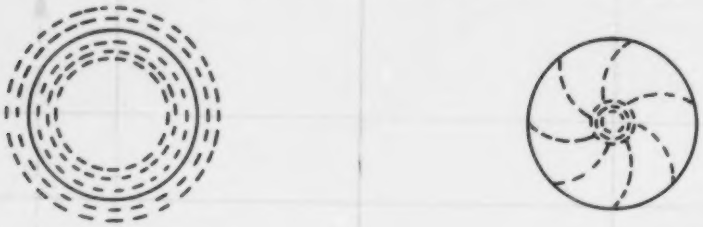

A. DIRECT IMPACT

B. REVERSE IMPACT

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Figure 17. Impact Failure Cracking Patterns (Typical).

No lifting or peeling of the acrylic, epoxy, or vinyl coatings was evident in wet-tape tests of these coatings on aluminum and steel. One of the polyester coatings (Plas No. 4009) lifted in spots from the aluminum, although it showed good adhesion on the steel. The other two polyester coatings showed good adhesion on both aluminum and steel. Tested panels coated with polyester and nylon powders are shown in Fig. 18, 19, and 20. The surface appearance of all the powder coatings was good. A slight orange-peel effect, typical of the powder coatings, was evident. The gloss varied from 66 to 96 for the 60-degree geometry. The polyamide coating showed a much lower gloss value because it was transparent. No difference in gloss was evident in the nylon applied by fluidized bed or powder spray. The epoxy coating provided the highest gloss of all the powder coatings tested.

Coating flexibility was determined by bending coated panels over a conical mandrel. The acrylic, epoxy, polyamide, nylon, and vinyl coatings and one polyester formulation showed excellent flexibility with no cracking visible when examined at 7X magnification. Polyester formulations Plas No. 4009 and 155-W-04130 showed film cracking.

Each of the powder coatings that showed good flexibility also showed good impact resistance on both aluminum and steel, except for the polyamide coating on aluminum. The coatings were tested up to impact levels of 8.5 j (75 in.-lb). Acrylic, epoxy, nylon, vinyl, and one polyester formulation showed no cracking, peeling, or lifting at this impact level for either reverse or direct impact. The polyamide coating on steel showed good impact resistance up to 8.5 j (75 in.-lb) but, on aluminum, this coating cracked at 3.4 j (30 in.-lb) on direct and reverse impact. Polyester formulation Plas No. 4009 failed due to film cracking at 2.3 j (20 in.-lb) for both direct and reverse impact on aluminum and steel. Polyester formulation 155-W-04130 cracked at less than 1.1 j (10 in.-lb) for both direct and reverse impact on aluminum and steel.

Resistance to heat, determined by exposure to 127 °C (260 °F) for 20 hr followed by 163 °C (325 °F) for 1 hr, caused various degrees of discoloration of the coatings. Only the epoxy and the nylon applied by fluidized bed showed more than slight discoloration due to heat exposure. The exposure to heat did not cause any other film defects.

Coatings were exposed to diester lubricating oil at 121 °C (250 °F) for 4 hr to determine the lubricating oil resistance of the coatings. After exposure, panels were examined for blistering and film softening. All of the coatings had some degree of discoloration after exposure. The epoxy, polyamide, polyester formulation Plas No. 4009, and nylon powder coatings showed no film defects after

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TABLE 16. POWDER COATINGS ON STEEL SUBSTRATES - SCREENING TEST RESULTS

Coating system	Surface appearance	Gloss (60°)	Anchorage (wet tape)	Flexibility (conical mandrel)	Impact resistance	Heat resistance	Lube oil resistance
Acrylic (154-W-04110)	Good	86	Pass	Pass	Pass 8.5 j (75 in.-lb)	Slight discoloration	Slight blistering; no film softening
Epoxy (Plas #5027)	Good	93	Pass	Pass	Pass 8.5 j (75 in.-lb)	Some discoloration	Some discoloration
Polyamide (Grilamid)	Good	30	Pass	Pass	Pass 8.5 j (75 in.-lb)	Slight discoloration	No film defects
Polyester (Plas #4009)	Good	80	Pass	Fail-film cracking at 0.4 in. dia	Fail @ 2.3 j (20 in.-lb)	Very slight discoloration	Some discoloration; no film defects
Polyester (155-W-04130)	Good	90	Pass	Fail-film cracking	Fail @ 2.3 j (20 in.-lb)	Very slight discoloration	Slight film softening; discoloration
Polyester (156-W-04102)	Good	91	Pass	Pass	Pass 8.5 j (75 in.-lb)	No discoloration	Slight blistering; no film softening
Nylon (Plas #1008)	Good	69	Pass	Pass	Pass 8.5 j (75 in.-lb)	Slight discoloration	Slight discoloration; no film defects
Nylon (Fluidized bed)	Good	66	Pass	Pass	Pass 8.5 j (75 in.-lb)	Discolored	Slight discoloration; no film defects
Vinyl	Good	78	Pass	Pass	Pass 8.5 j (75 in.-lb)	No discoloration	Discoloration; complete film softening

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TABLE 17. POWDER COATINGS ON ALUMINUM SUBSTRATES - SCREENING TEST RESULTS

Coating system	Surface appearance	Gloss (60°)	Anchorage (wet tape)	Flexibility (conical mandrel)	Impact resistance	Heat resistance	Lube oil resistance
Acrylic (Hi Temp) (154-W-04110)	Good	84	Pass	Pass	Pass 8.5 j (75 in.-lb)	Very slight discoloration	Discoloration; no film defects
Acrylic (Lo Temp) (154-W-04110)	Good	90	Pass	Pass	Pass 8.5 j (75 in.-lb)	Very slight discoloration	Discoloration; slight film softening
Epoxy (Plas #5027)	Good	96	Pass	Pass	Pass 8.5 j (75 in.-lb)	Some discoloration	Some discoloration; no film defects
Polyamide (Grilamid)	Good	43	Pass	Pass	Fail @ 3.4 j (30 in.-lb)	Slight discoloration	Slight discoloration; no film defects
Polyester (Plas #4009)	Good	79	Lifted in spots	Fail-film cracking	Fail @ 2.3 j (20 in.-lb)	Very slight discoloration	Slight discoloration; no film defects
Polyester (Hi Temp) (155-W-04130)	Good	86	Pass	Slight film cracking	Fail @ 0.2 j (2 in.-lb)	Very slight discoloration	Discoloration; film softening
Polyester (Lo Temp) (155-W-04130)	Good	84	Pass	Slight film cracking	Fail @ 1.1 j (10 in.-lb)	Very slight discoloration	Discoloration; film softening
Polyester (Hi Temp) (156-W-04102)	Good	88	Pass	Pass	Pass 8.5 j (75 in.-lb)	Very slight discoloration	Discoloration; slight film softening
Polyester (Lo Temp) (156-W-04102)	Good	88	Pass	Pass	Marginal at 6.8 j (60 in.-lb)	Very slight discoloration	Discoloration; slight film softening
Nylon (Plas #1008)	Good	68	Pass	Pass	Pass 8.5 j (75 in.-lb)	Slight discoloration	Slight discoloration; no film defects
Nylon (Fluidized bed)	Good	68	Pass	Pass	Pass 8.5 j (75 in.-lb)	Discoloration	Slight discoloration; no film defects
Vinyl	Good	66	Pass	Pass	Pass 8.5 j (75 in.-lb)	No discoloration	Discoloration; complete film softening

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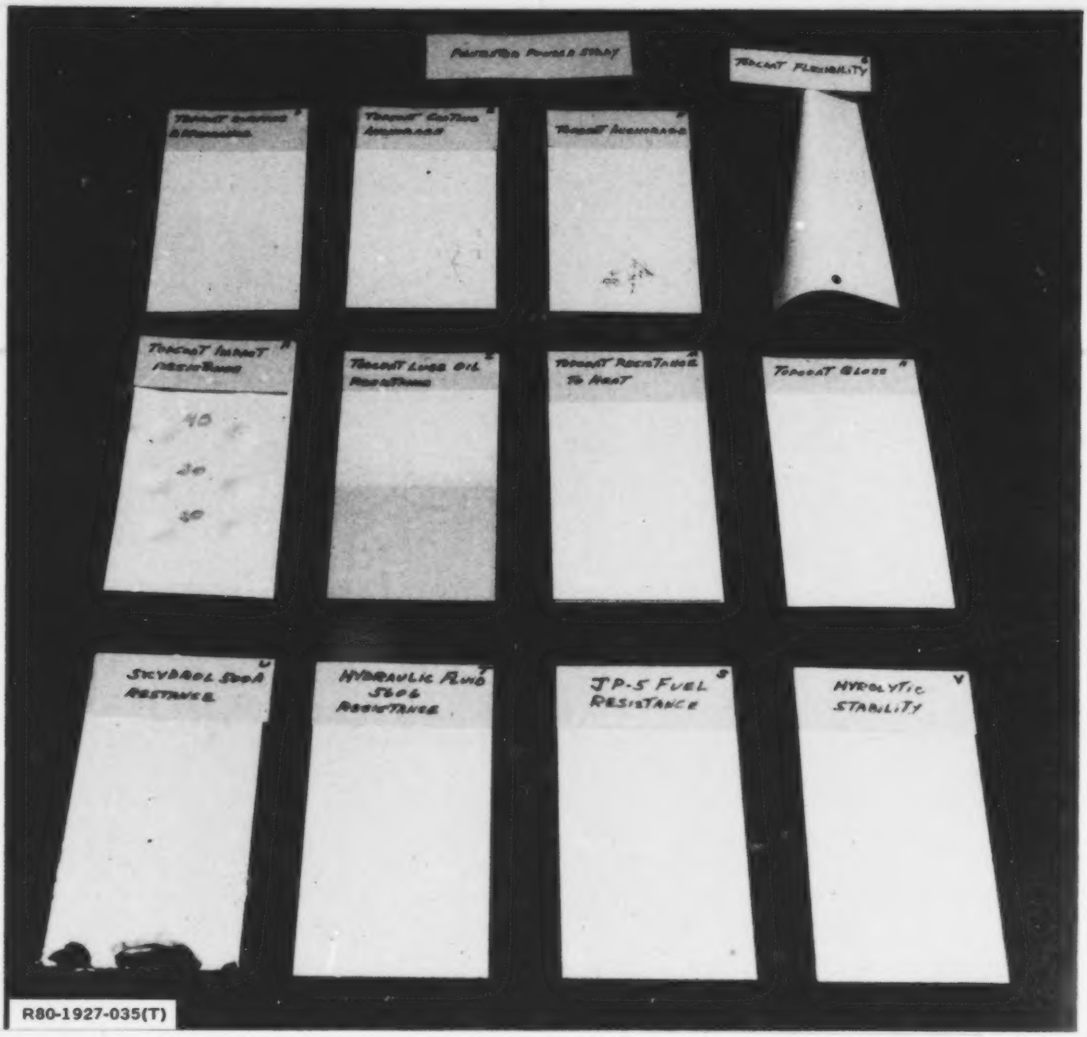
POLYESTER POWDER STUDY
TOPCOAT FLEXIBILITY
TOPCOAT IMPACT RESISTANCE
40
30
20
TOPCOAT LUBE OIL RESISTANCE
TOPCOAT RESISTANCE TO HEAT
TOPCOAT GLOSS
SKYDROL 500A RESISTANCE
HYDRAULIC FLUID 5606 RESISTANCE
JP-5 FUEL RESISTANCE
HYDROLYTIC STABILITY

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Figure 18. Tested Panels Spray-Coated With Polyester Powder.

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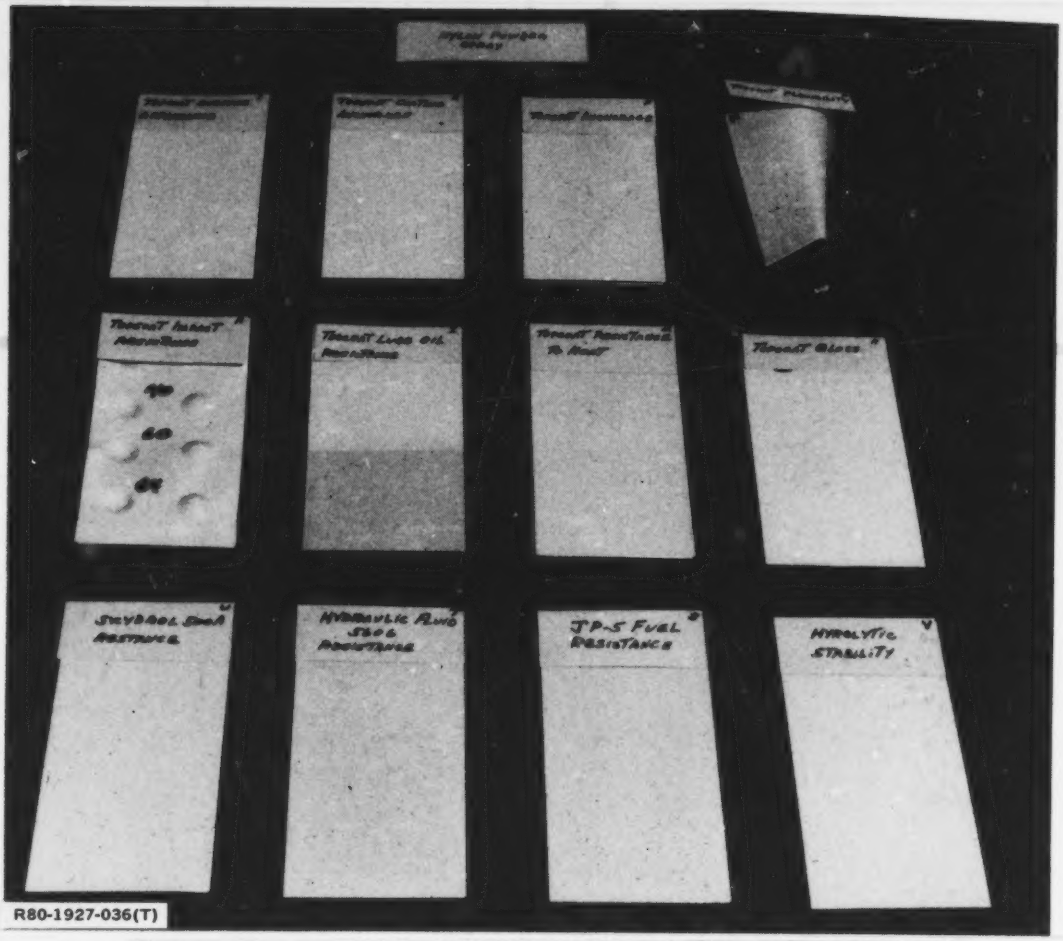
SKYDROL 500A RESISTANCE
HYDRAULIC FLUID 5606 RESISTANCE
JP-5 FUEL RESISTANCE
HYDROLYTIC STABILITY
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Figure 19. Tested Panels Spray-Coated With Nylon Powder.

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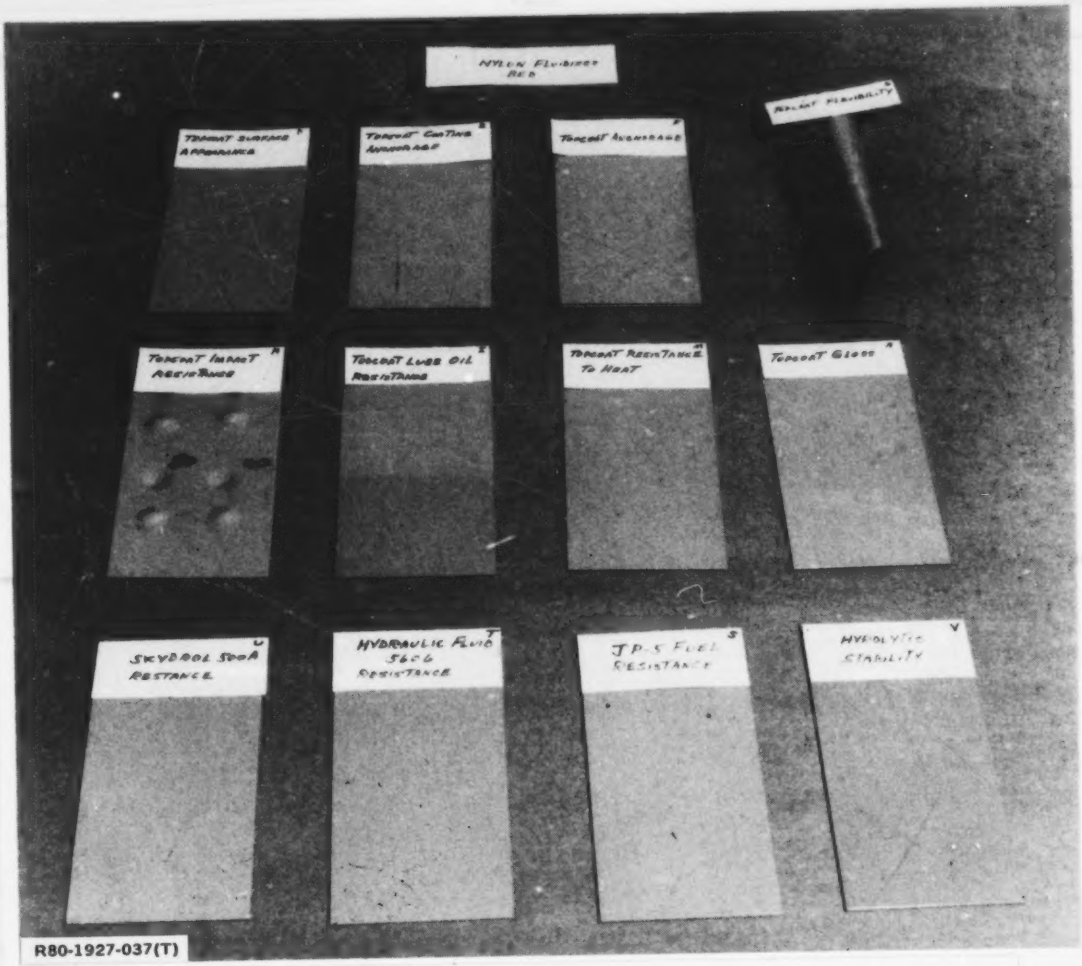

NYLON FLUIDIZED BED
TOPCOAT SURFACE APPEARANCE
TOPCOAT COATING ADHERANCE
TOPCOAT ADHERANCE
TOPCOAT FLEXIBILITY
TOPCOAT IMPACT RESISTANCE
TOPCOAT LUBE OIL RESISTANCE
TOPCOAT RESISTANCE TO HEAT
TOPCOAT GLOSS
SKYDROL 500A RESTANCE
HYDRAULIC FLUID 5606 RESISTANCE
JP-5 FUEL RESISTANCE
HYDROLYTIC STABILITY
R80-1927-037(T)

Figure 20. Tested Panels Fluidized Bed-Coated With Nylon Powder.

lubricating oil exposure on either aluminum or steel substrates. The acrylic and polyester formulation 156-W-04102 showed slight blistering or slight film softening, depending on substrate and cure temperature. A greater degree of film softening occurred with polyester formulation 155-W-04130. The lubricating oil exposure caused complete film softening of the vinyl coating which remained soft and tacky in the exposed areas.

Based on these preliminary screening tests, five powder coatings were selected for further evaluation. The selected coatings provided excellent performance in the screening tests. The selected coatings include acrylic (high-temperature cure), epoxy, polyester formulation 156-W-04102 (high-temperature cure) and nylon applied to aluminum and steel substrates by electrostatic spray, polyamide on steel applied by electrostatic spray, and nylon on aluminum and steel by fluidized-bed application.

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Water-Borne Maskants

Candidate water-borne maskants were subjected to preliminary screening tests to evaluate appearance, peelability, etchant resistance, line definition, and scribability. Each of the maskants had a uniform appearance with no pin-holing or bubbling. The thickness of the Adcoat and Dee Aircraft maskants was 12 mils (0.0005 in.) while that of the Turco material was 8 mils (0.0003 in.).

Masked aluminum panels were scribed and etched in sodium hydroxide etchant. Each material scribed easily. The Adcoat and Turco masks showed good line definition, while that of Dee Aircraft was poor. The peelability and etchant resistance of the Adcoat mask were good. Since only a few test panels of each material were provided by the manufacturers, no further testing was conducted. These preliminary tests do show, however, that these candidate water-borne maskants have potential as replacements for organic-solvent maskants.

Final Characterization Tests

Final characterization tests of the selected water-borne paints and powder coatings included additional fluid resistance tests and hydrolytic stability evaluation. A performance comparison of the water-borne paints and powder coatings is shown in Table 18. The performance of the water-borne paints is comparable to that of the organic solvent paint control with respect to most properties; the bake-cure systems were slightly better than the air-dry systems. The selected powder coatings performed as well as or better than the organic-solvent paint control in most cases. The nylon powder coatings provided the best overall performance. Tested panels are shown in Fig. 21 through 24.

The selected coatings showed good appearance, adhesion, and flexibility. Gloss ranged from 30 for the clear polyamide powder coating to 95 for the epoxy powder coating. The impact resistance of the selected coatings was very good with no cracking or peeling on either direct or reverse impact. Exposure to heat and lubricating oil caused varying degrees of discoloration in all of the coatings tested. The lubricating oil also caused film softening in the two bake-cure paint systems and in the polyester powder coating.

The resistance of the selected coatings to three fluids (JP-5 fuel, HF-5606 hydraulic fluid and Skydrol 500 hydraulic fluid) was determined by immersing coated panels in each of the fluids for 7 days at room temperature. The coatings were virtually unaffected by the JP-5 fuel and the HF-5606 fluid except for slight discoloration of the water-borne paints. The epoxy-polyurethane control system did show slight blistering following HF-5606 fluid exposure, although no film softening or loss of adhesion resulted. The effect of Skydrol 500 fluid exposure was much more severe; film softening and loss of adhesion occurred in two of the paint systems and one of the powder coatings. These coatings remained soft after removal from the fluid. The polyester powder coating showed moderate film softening and the acrylic powder coating showed very slight film softening as a result of Skydrol 500 fluid exposure. The Skydrol 500 fluid had no effect on the LP-3724 paint system and the polyamide and nylon powder coatings.

The hydrolytic stability test, designed to show the effect of high temperature and humidity on the coating systems, had no effect on the epoxy-polyurethane control system. The LP-3724 paint system and the acrylic, polyester, and nylon powder coatings were also unaffected by the hydrolytic stability exposure. Loss of adhesion of the epoxy and polyamide powder coatings resulted from this exposure. Two paint systems, Aquathane II and Aqualure 634-W-804, had film softening and loss of adhesion as a result of the hydrolytic stability exposure.

The loss of gloss caused by applying a primer under the topcoat was also determined for the paint systems under test. The topcoat was applied to primed and unprimed panels and the gloss determined on each. The difference in gloss between the primed and unprimed panels ranged from one to ten units; the greatest loss of gloss occurred with the Aqualure 634-W-804 system.

TABLE 18. FINAL CHARACTERIZATION TESTS: REPLACEMENT COATINGS FOR ORGANIC SOLVENT PAINTS

Coating System	Coating Thickness, Mils	Surface Appearance	Gloss (60°)	Anchorage (Wet Tape)	Flexibility (Conical Mandrel)	Impact Resistance	Heat Resistance	Lube Oil Resistance	Resistance To Fluids			Hydrolytic Stability
									JP-5	HF-5606	Skydrol 500	
Control System												
Epoxy Primer Polyurethane Topcoat	.0015	V Sl Mottling	89	Good	Pass	Pass 8.5 j (75 in.-lb)	V Sl discoloration	No film defects; Sl discoloration	No effect	Sl blistering	Film softened; loss of adhesion	No film softening or loss of adhesion; Sl discoloration
Air-Dry Paint Systems												
Epoxy Primer Aquathane II Topcoat	.0015	Sl Mottling	54	Good	Pass	Pass 8.5 j (75 in.-lb)	Discoloration	No film defects; discoloration	No film defects; Sl discoloration	No film defects; Sl discoloration	Film softened; loss of adhesion	Film softening; some loss of adhesion; discoloration
Bake Cure Paint Systems												
Aqualure 631-L-128 Primer 634-W-804 Topcoat	.005	Good	88	Good	Pass	Pass 8.5 j (75 in.-lb)	V Sl discoloration	Film softening; Sl discoloration	No film defects; Sl discoloration	No film defects; Sl discoloration	Film softened; loss of adhesion	Film softening; loss of adhesion
LP-3778 Primer LP-3724 Topcoat	.0025	Good, V Sl seeding & orange peel	69	Sl lifting along scribes	Pass	Pass 8.5 j (75 in.-lb)	No effect	Sl film softening; discoloration	No effect	No effect	No effect	No effect
Powder Coatings												
Acrylic (Hi Temp) (154-W-04110) On Aluminum & Steel	.0015 (Al) .001 (Steel)	Good	85	Good	Pass	Pass 8.5 j (75 in.-lb)	V Sl discoloration	No film softening; discoloration; (V Sl blistering on steel)	No effect	No effect	V Sl film softening	No effect
Epoxy (Plas =5027) On Aluminum & Steel	.0015 (Al) .0015 (Steel)	Good	94	Good	Pass	Pass 8.5 j (75 in.-lb)	Some discoloration	Some discoloration	No effect	No effect	Film softened; complete loss of adhesion	Blistering; loss of adhesion; some discoloration
Polyamide (Grilamid) On Steel Only	.005	Good	30	Good	Pass	Pass 8.5 j (75 in.-lb)	Sl discoloration	No film defects	No effect	No effect	No effect	Loss of adhesion
Polyester (Hi Temp) (156-W-04102) On Aluminum & Steel	.00175 (Al) .0015 (Steel)	Good	89	Good	Pass	Pass 8.5 j (75 in.-lb)	V Sl discoloration	Sl film softening; discoloration	No effect	No effect	Moderate film softening	No effect
Nylon (Powder Spray & Fluidized Bed) (Plas =1008 & LNP & RILSAN) On Aluminum & Steel	Powder .00525 (Al) Powder .004 (Steel) Nylon 3 dip .0055 (Al) Nylon 2 dip .0103 (Steel)	Good	68	Good	Pass	Pass 8.5 j (75 in.-lb)	Sl discoloration	No film defects; Sl discoloration	No effect	No effect	No effect	No film defects; Sl discoloration

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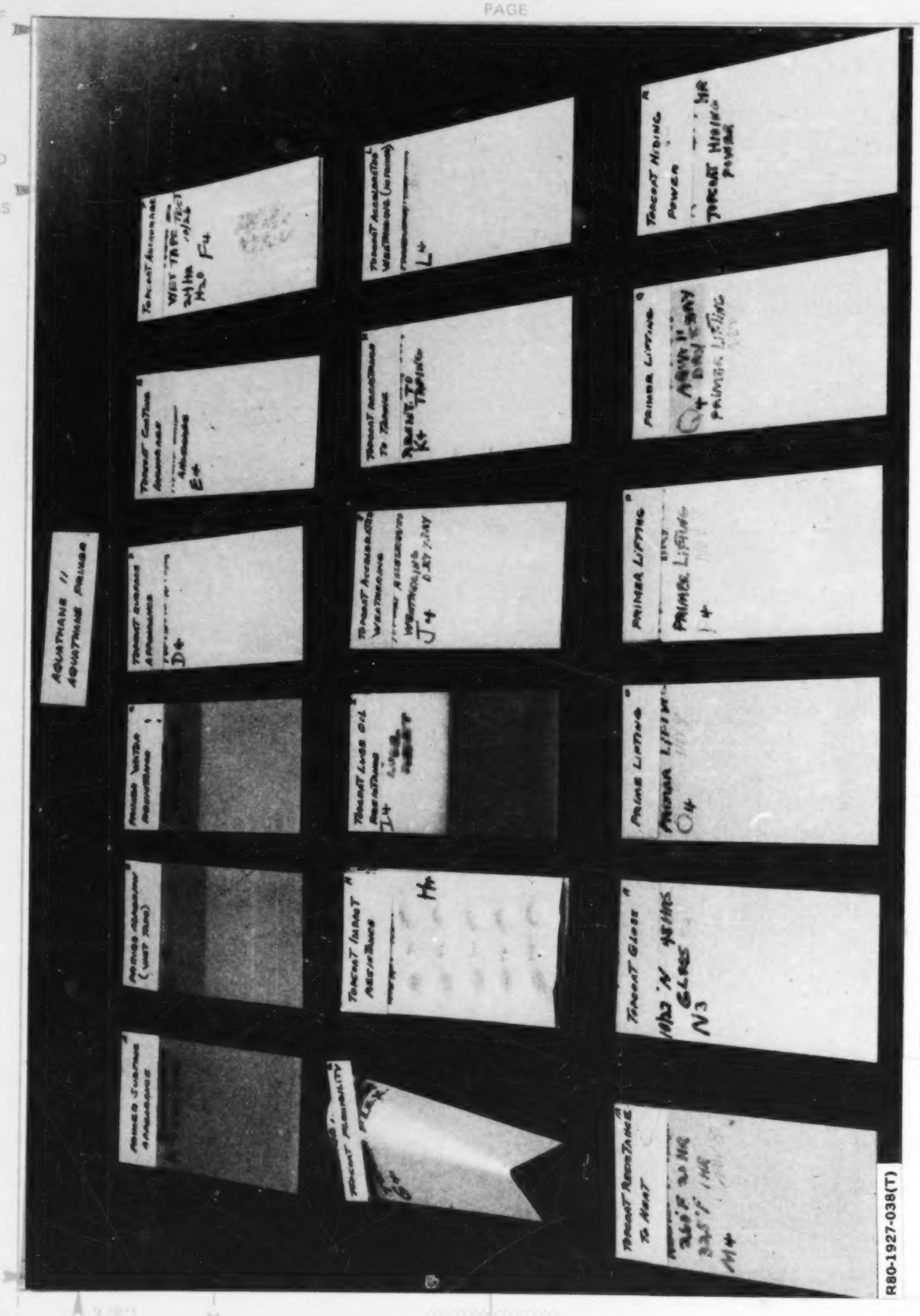
AQUATHANE II
AQUATHANE PRIMER
TOPCOAT ADHESION
WET TAPE TEST
24 HR H2O
F+
TOPCOAT HIDING POWER
TOPCOAT HIDING POWER
PRIMER LIFTING
PRIMER LIFTING
PRIMER LIFTING
TOPCOAT GLOSS
N3
TOPCOAT IMPACT RESISTANCE
TOPCOAT RESISTANCE TO HEAT
M+
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Figure 21. Results of Final Characterization Tests of Panels Coated With Aquathane Primer/Aquathane II Topcoat.

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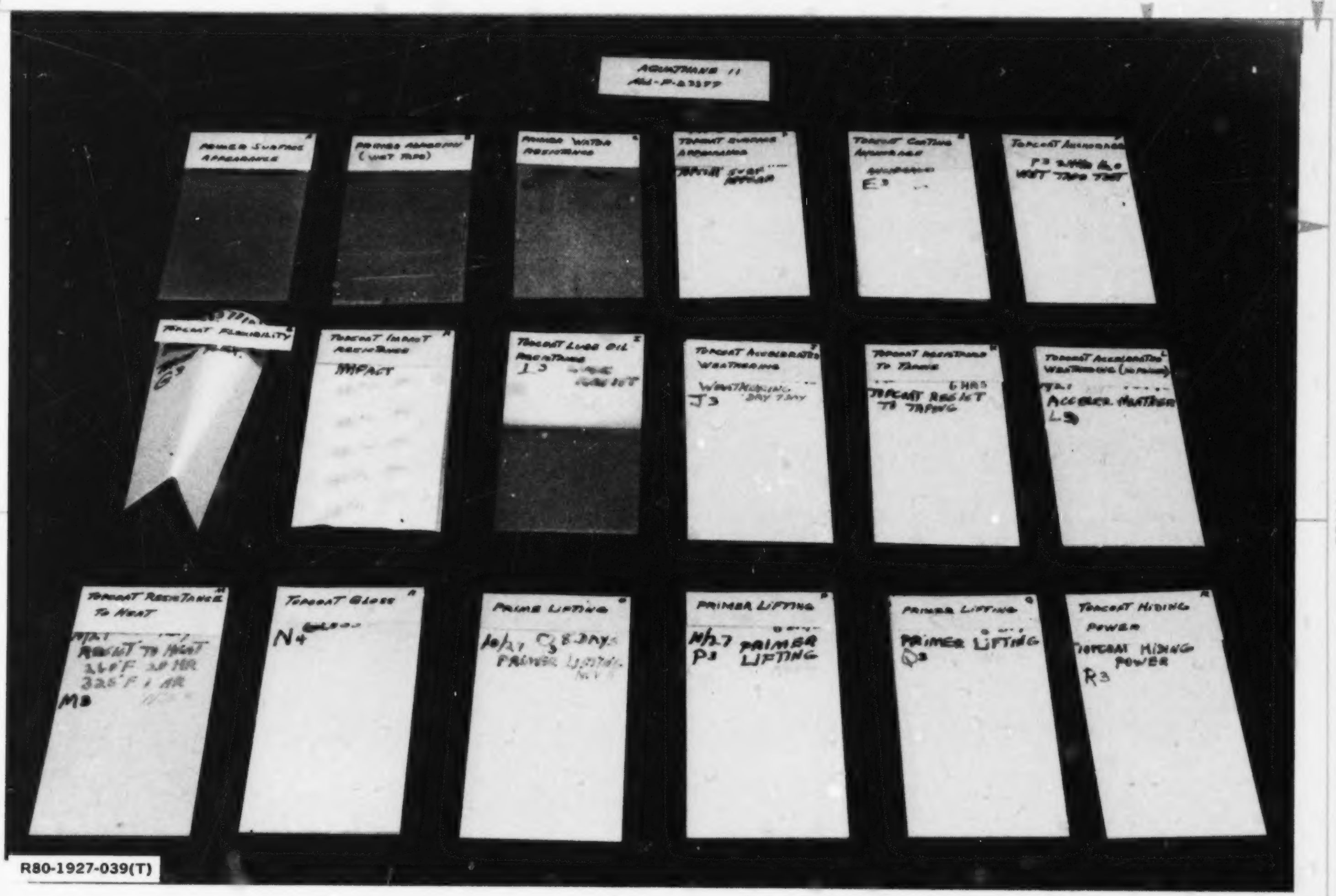
AQUATHANE II
MIL-P-23377
PRIMER SURFACE APPEARANCE
PRIMER ADHESION (WET TAPE)
PRIMER WATER RESISTANCE
TOPCOAT SURFACE APPEARANCE
TOPCOAT COATING APPEARANCE
E3
TOPCOAT ADHESION
P3 24HR ADH WET TAPE TEST
TOPCOAT FLEXIBILITY
G3
TOPCOAT IMPACT RESISTANCE
IMPACT
TOPCOAT LUBE OIL RESISTANCE
I3
TOPCOAT ACCELERATED WEATHERING
WEATHERING
J3
TOPCOAT RESISTANCE TO TAPING
6 HRS
TOPCOAT RESIST TO TAPING
TOPCOAT ACCELERATED WEATHERING
ACCELER. WEATHER
L3
TOPCOAT RESISTANCE TO HEAT
RESIST TO HEAT
260°F 20 HR
325°F 1 HR
M3
TOPCOAT GLOSS
N4
PRIME LIFTING
10/27 C3 8 DAYS
PRIMER LIFTING
PRIMER LIFTING
11/27 P3 PRIMER LIFTING
PRIMER LIFTING
PRIMER LIFTING
Q3
TOPCOAT HIDING POWER
TOPCOAT HIDING POWER
R3
R80-1927-039(T)

Figure 22. Results of Final Characterization Tests of Panels Coated With MIL-P-23377 Primer/Aquathane II Topcoat.

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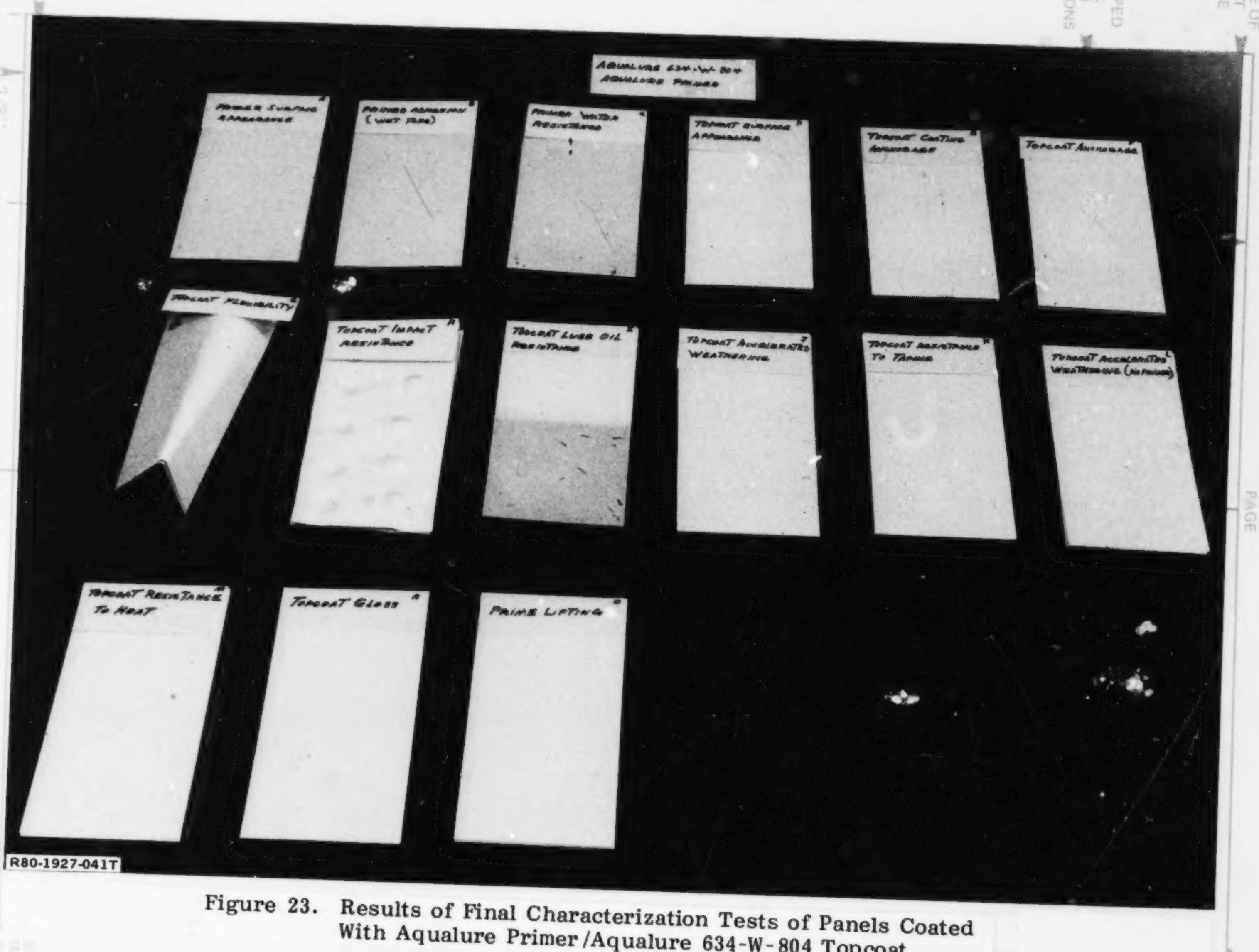
AQUALURE 634-W-804
AQUALURE PRIMER
PRIMER SURFACE APPEARANCE
PRIMER ADHESION (WET TAPE)
PRIMER WATER RESISTANCE
TOPCOAT SURFACE APPEARANCE
TOPCOAT COATING ANCHORAGE
TOPCOAT ANCHORAGE
TOPCOAT FLEXIBILITY
TOPCOAT IMPACT RESISTANCE
TOPCOAT LUBE OIL RESISTANCE
TOPCOAT ACCELERATED WEATHERING
TOPCOAT RESISTANCE
TOPCOAT ACCELERATED WEATHERING (NO PRIMER)
TOPCOAT RESISTANCE TO HEAT
TOPCOAT GLOSS
PRIME LIFTING
R80-1927-041T

Figure 23. Results of Final Characterization Tests of Panels Coated With Aqualure Primer/Aqualure 634-W-804 Topcoat.

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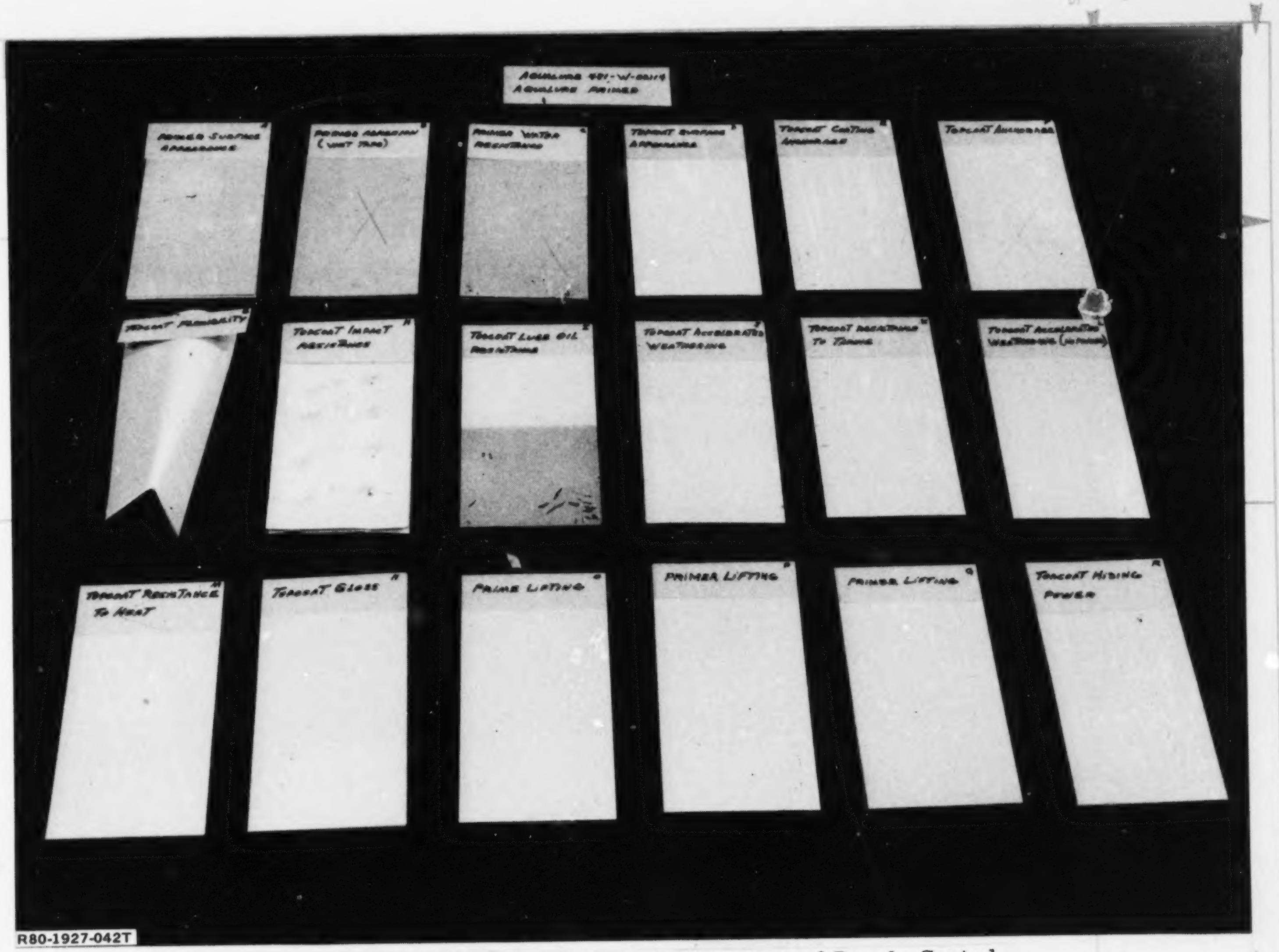
Aqualure 481-W-02114
Aqualure Primer
Primer Surface Appearance
Primer Adhesion (Wet Tape)
Primer Water Resistance
Topcoat Surface Appearance
Topcoat Coating Adhesion
Topcoat Adhesion
Topcoat Flexibility
Topcoat Impact Resistance
Topcoat Lube Oil Resistance
Topcoat Accelerated Weathering
Topcoat Resistance To Fluids
Topcoat Accelerated Weathering
Topcoat Resistance To Heat
Topcoat Gloss
Prime Lifting
Primer Lifting
Primer Lifting
Topcoat Hiding Power
R80-1927-042T

Figure 24. Results of Final Characterization Test of Panels Coated With Aqualure Primer/Aqualure 481-W-02114 Topcoat.

Long-Term, Outdoor Exposure

The selected water-borne paints and powder coatings applied to aluminum and steel test panels were subjected to 12 months of outdoor (southerly) exposure at Grumman's Test Site located in the U.S. Coast Guard Station at Fire Island, N.Y. (Fig. 25). Results of tests conducted on coated panels exposed for 3, 6, 9, and 12 months are compared to those for unexposed panels in Table 19. The epoxy/polyurethane control system, as well as the Aquathane II and Aqualure 634-W-804 paint systems and the acrylic powder coating, resisted the 12-month weathering period rather well. Compared to the unexposed specimens, the coated panels experienced no significant loss in properties. The epoxy powder coating on aluminum test panels also showed no significant loss in properties despite chalking and a severe loss of gloss; however, this coating on steel test panels had severe corrosion after 6 months' exposure. Although the oven-cured, water-borne paint system (LP 3778 primer and LP 3724 topcoat) showed no corrosion after 12 months' exposure, its adhesion, flexibility, and impact resistance decreased after 3 months exposure. Polyester 156 coating applied to aluminum test panels experienced a slight decrease in impact resistance. When applied to steel test panels, however, the Polyester 156 coating showed evidence of corrosion around the panel edges after 3 months' exposure, with a slight decrease in flexibility and impact resistance. Both the nylon powder spray and fluidized-bed coating experienced a sharp loss in gloss and a decrease in flexibility and impact resistance, even though no loss of adhesion occurred. The substrate used did not appear to affect the wearability of these coatings. The acrylic and polyamide powder spray coatings showed evidence of corrosion after 3 months' exposure and severe corrosion after 6 months' exposure, and were unable to undergo testing after 12 months' exposure (corrosion was too severe).

Economic and Environmental Evaluation

Water-borne paints and powder coatings provide a means of eliminating the pollution control problem associated with organic solvent paints. Solvent collection systems are not required for either coating type. Water-borne paints are formulated for either air-drying or elevated-temperature curing. Powder coatings can be applied either by an electrostatic spray or a fluidized bed. Different equipment is required for each of these techniques, which have various advantages relative to the solvent-borne systems and to each other.

Air-drying, water-borne paint systems require the same application equipment as solvent-borne systems, except that a solvent collection system is not required. Thus, an existing facility can be converted directly for use with water-borne paints. The cost of a new installation would be less for the water-borne system by the amount of the solvent collection system. Elevated-temperature-curing water-borne paint systems have the same equipment considerations as the air-drying systems, with the added requirement that adequate oven capacity be available to cure the painted parts. Pretreatment of the substrate is essentially the same as that for each type of paint system.

An electrostatic powder spray facility could consist basically of a manual electrostatic powder spray gun with powder reservoir and power supply, a conventional spray booth, and a small curing oven, or it could involve an automated line with multiple spray guns and a powder recovery system capable of 98% powder utilization. The installation cost would depend on the complexity of the system used. Operation of a powder coating line is cost-competitive with solvent-based paint application. Material costs are lower because powder coatings are 100% solid and complete powder utilization is possible with efficient recovery systems. Fluidized bed application of powder coatings requires a fluidizing tank and a pre-heating/curing oven. The advantages are the same: high material utilization, lower material cost for the same resin system, and no solvent recovery system requirement.

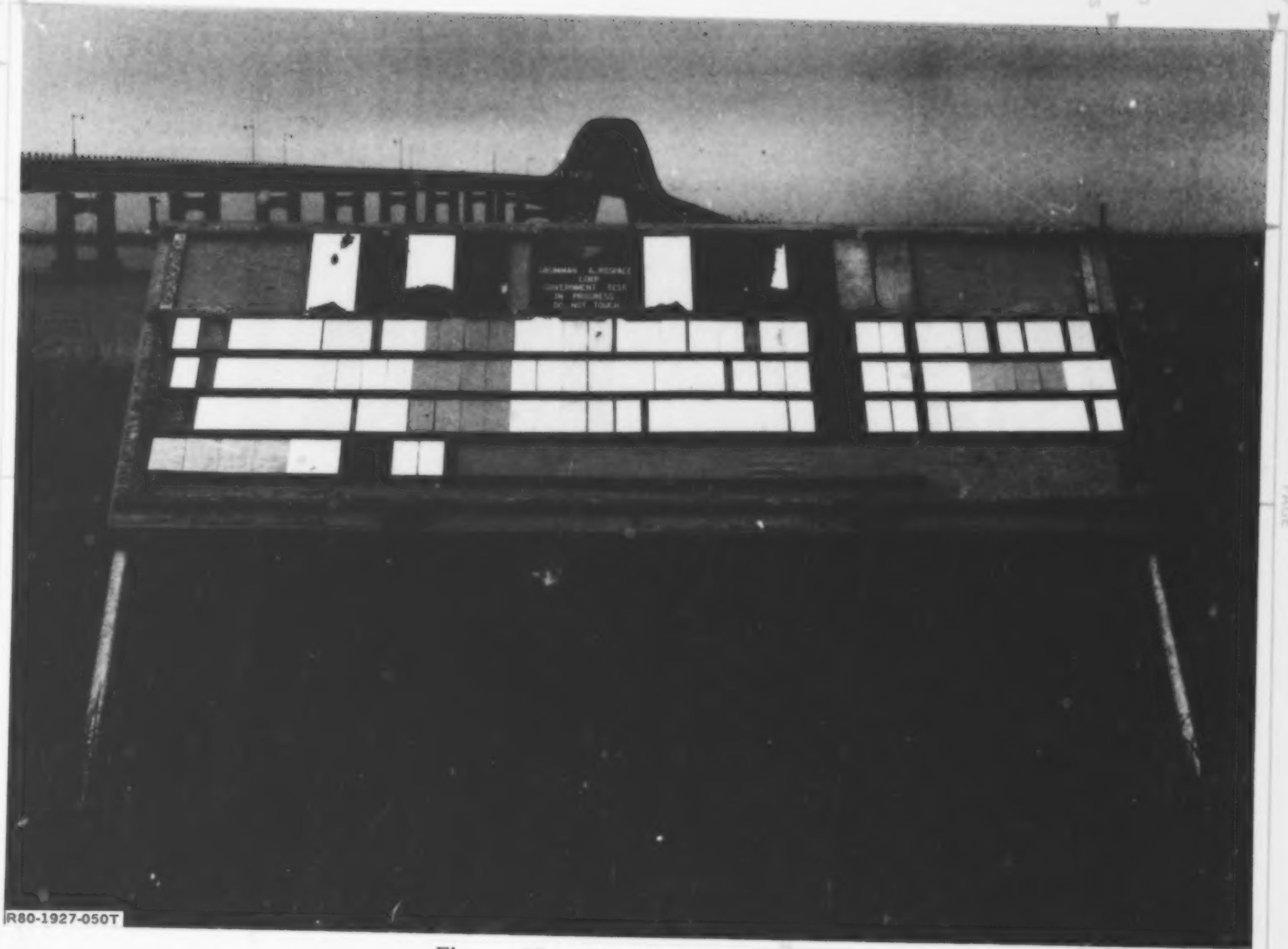
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Figure 25. Fire Island Test Stand.

TABLE 19. EFFECT OF OUTDOOR WEATHERING ON PERFORMANCE OF PROTECTIVE COATING SYSTEMS

Coating System	Substrate	Exposure Time, Months	Performance Evaluation Factors					
			Appearance	Corrosion	Gloss	Adhesion	Flexibility	Impact Resistance, J (in.-lb)
Epoxy/Polyurethane Paint (Control)	Aluminum	0*	Good	–	89	No lifting	Pass	Pass—8.5 (75) Dir & Indir
		3	Good	None	89	Slight lifting along scribes	Pass	Pass—8.5 (75) Dir & Indir
		6	Good	None	88	No lifting	Pass	Fail—3.4 (30) Dir & Fail—1.1 (10) Indir
		9	Good	None	80	–	Pass	Fail—6.8 (60) Dir & Fail—8.5 (75) Indir
		12	Good	None	–	No lifting	Pass	Fail—6.8 (60) Dir & Fail—3.4 (30) Indir
Acrylic Powder Spray	Steel	0*	Good	–	85	Pass	Pass	Pass—8.5 (75), Dir & Indir
		3	Good	Some on edges & face of panel	95	One spot on scribe	Much fine cracking	Fail—8.5 (75) Dir, Fail—4.5 (40) Indir
		6	Blistered	Spotty all over	72	Lifting in corroded areas; very slight lifting elsewhere	Not tested	Not tested
		9	Edges blistered	Spotty all over	Not tested	Not tested	Lifting along entire length	Fail—3.4 (30) Dir & Fail—2.3 (20) Indir
		12	Very severely corroded	All over	Not tested	Not tested	Not tested	Not tested
Acrylic Powder (High-Temp Cure)	Aluminum	0*	Good	–	85	No lifting	Pass	Pass—8.5 (75) Dir & Indir
		3	Good	None	73	No lifting	Very slight cracking	Pass—8.5 (75) Dir & Indir
		6	Very good	None	79	No lifting	Very slight cracking	Fail—8.5 (75) Dir & Pass—8.5 (75) Indir
		9	Very good	None	80	No lifting	Pass	Fail—7.9 (70) Dir & Fail—2.3 (20) Indir
		12	Very good	None	63	No lifting	Very fine cracks (3/4 in.)	Fail—7.3 (65) Dir & Fail—8.5 (75) Indir
Aquathane II Paint with Epoxy Primer	Aluminum	0*	Good	–	54	Good	Pass	Pass—8.5 (75) Dir & Indir
		3	Good	None	79	Slight lifting along scribes	Pass	Pass—8.5 (75) Dir & Indir
		6	Good; very slightly yellowed	None	59	Very slight lifting along scribes	Pass	Fail—2.3 (20) Dir & Fail—3.4 (30) Indir
		9	Good	None	41	Lifting along scribes; 50% lifting between lines	Pass	Fail—3.4 (30) Dir & Indir
		12	Good	None	23	Lifting along scribes	Pass	Fail—3.9 (35) Dir & Fail—2.8 (25) Indir
Aqualure 634-W-804 Paint	Aluminum	0*	Good	–	88	No lifting	Pass	Pass—8.5 (75) Dir & Indir
		3	Good	None	79	No lifting	Pass	Pass—8.5 (75) Dir & Indir
		6	Good	None	76	No lifting	Pass	Pass—8.5 (75) Dir & Indir
		9	Good	None	80	No lifting	Pass	Pass—8.5 (75) Dir & Indir
		12	Good	One rust spot	–	No lifting	Pass	Fail—8.5 (75) Dir & Indir
LP3778 Primer/ LP3724 Topcoat	Aluminum	0*	Good	–	69	Slight lifting along scribes	Pass	Pass—8.5 (75) Dir & Indir
		3	Slightly dull	None	49	Lifting along scribes	Cracked & peeled from substrate	Fail—1.1 (10) Dir & Indir
		6	Good; some blistering along edges	None	28	Spot lifting along scribes	Cracked & peeled from substrate	Fail—2.3 (25) Dir & Fail—1.1 (10) Indir

*Unexposed (control)

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(continued)

TABLE 19. EFFECT OF OUTDOOR WEATHERING ON PERFORMANCE OF PROTECTIVE COATING SYSTEMS (Continued)

Coating System	Substrate	Exposure Time, Months	Performance Evaluation Factors					
			Appearance	Corrosion	Gloss	Adhesion	Flexibility	Impact Resistance, J (in.-lb)
		9	Slightly dull	None	43	Lifting along scribes; small spot lifting between lines	Pass	Fail–1.1 (10) Dir & Fail–0.56 (5) Indir
		12	Dull	None	16	Lifting along scribes; small spot lifting between lines	Cracking along entire length	Fail–1.1 (10) Dir & Fail–0.23 (2) Indir
Epoxy Powder Spray	Steel	0*	Good	–	94	Pass	Pass	Pass–8.5 (75) Dir & Indir
		3	Dull; color shift	None	41	No lifting	Much fine cracking	Fail–4.5 (40) Dir & Fail–1.1 (10) Indir
		6	Dull; yellowed; blistered; peeling	Very heavy from edges	6	Not tested	Not tested	Not tested
		9	Very severely corroded	Severe; almost no paint left	–	Not tested	Not tested	Not tested
		12	Very severely corroded	Severe; almost no paint left	–	Not tested	Not tested	Not tested
Epoxy Powder Spray	Aluminum	0*	Good	–	94	No lifting	Pass	Pass–8.5 (75) Dir & Indir
		3	Yellowed, dull	None	15	No lifting	Slight cracking	Pass–8.5 (75) Dir & Indir
		6	Yellowed, dull	None	7	No lifting	Very slight cracking	Fail–8.5 (75) Dir & Indir
		9	Yellowed, dull	None	4	No lifting	Fine cracking to 3/4 in.	Fail–8.5 (75) Dir & Fail–6.8 (60 Indir
		12	Yellowed, dull	Small rust spots	6	No lifting	Cracking to 3/4 in, very fine	Pass–8.5 (75) Dir & Indir
Nylon Powder Spray	Aluminum	0*	Good	–	68	Pass	Pass	Pass–8.5 (75) Dir & Indir
		3	*Dull, color shift*	*None*	53	*No lifting*	Several large cracks	Fail–2.3 (20) Dir & Fail–1.1 (10) Indir
		6	Good	None	25	No lifting	Cracked & peeled from substrate	Fail–2.3 (20) Dir & Fail–1.1 (10) Indir
		9	Whitened, dull	None	6	Very slight lifting along scribes	Very fine cracking to 3/4 in.	Fail–1.1 (10) Dir & Fail–3.4 (30) Indir
		12	Whitened, dull	None	8	No lifting	Very fine cracking to 3/4 in.	Fail–1.1 (10) Dir & Indir
Nylon Powder Spray	Steel	0*	Good	–	68	Pass	Pass	Pass–8.5 (75) Dir & Indir
		3	Dull, color shift	None	41	No lifting	Fine cracking	Fail–4.5 (40) Dir & Fail–1.1 (10) Indir
		6	Good, slightly dull	None	21	No lifting	Much fine cracking; some large cracking	Fail–4.5 (40) Dir & Fail–1.1 (10) Indir
		9	Whitened, dull	None	15	No lifting	Cracking along entire length	Fail–3.4 (30) Dir & Fail–4.5 (40) Indir
		12	Whitened, dull	Rust on edge of panel	8	No lifting	Cracking along entire length	Fail–4.5 (40) Dir & Fail–1.1 (10) Indir

*Unexposed (control)

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TABLE 19. EFFECT OF OUTDOOR WEATHERING ON PERFORMANCE OF PROTECTIVE COATING SYSTEMS (Continued)

Coating System	Substrate	Exposure Time, Months	Performance Evaluation Factors					
			Appearance	Corrosion	Gloss	Adhesion	Flexibility	Impact Resistance, J (in.-lb)
Nylon Fluidized Bed (2 Dips)	Steel	0*	Good	–	69	Pass	Pass	Pass–8.5 (75) Dir & Indir
		3	Slight dulling	None	31	No lifting	Heavy cracking	Fail–4.5 (40) Dir & Fail–1.1 (10) Indir
		6	Slight dulling	None	22	No lifting	Cracked & peeled from substrate	Fail–3.4 (30) Dir & Fail–1.1 (10) Indir
		9	Dull	None	6	No lifting	Lifting along entire length	Fail–2.3 (20) Dir & Fail–1.1 (10) Indir
		12	Dull	Rust on edge of of panel	14	No lifting	Cracking along entire length	Fail–3.4 (30) Dir & Fail–1.1 (10) Indir
Nylon Fluidized Bed (3 Dips)	Aluminum	0*	Good	–	68	Pass	Pass	Pass–8.5 (75) Dir & Indir
		3	Slight dulling	None	45	Very slight lifting along scribes	Cracked	Fail–2.3 (20) Dir & Fail–1.1 (10) Indir
		6	Good	None	45	No lifting	Cracked	Fail–2.3 (20) Dir & Fail–1.1 (10) Indir
		9	Dull	None	19	Very slight lifting along scribes	Cracked along entire length	Fail–1.7 (15) Dir & Fail–1.1 (10) Indir
		12	Dull	None	25	No lifting	Cracked along entire length	Fail–1.1 (10) Dir & Indir
Polyester 156 Powder Spray	Steel	0*	Good	–	89	No lifting	Pass	Pass–8.5 (75) Dir & Indir
		3	Good	On edges; paint blistered	81	No lifting	Some cracking	Fail–6.8 (60) Dir & 1.1 (10) Indir
		6	Good	On edges; paint blistered	81	No lifting	Very fine cracking	Fail–7.9 (70) Dir & 3.4 (30) Indir
		9	Good	On edges; paint blistered	72	Very slight lifting along scribes	Lifting at extreme ends; cracking in middle	Fail–3.4 (30) & 1.1 (10) Indir
		12	Edges blistered; center very good	Advancing from edges to center	Not tested	Lifting along scribes & between lines	Lifting at extreme ends; cracking in center	Fail–7.3 (65) Dir & 1.1 (10) Indir
Polyester 156 Powder Spray	Aluminum	0*	Very good	–	88	No lifting	Pass	Pass–8.5 (75) Dir & Indir
		3	Very good	None	82	No lifting	Slight cracking	Fail–4.5 (40) Dir & Fail–1.1 (10) Indir
		6	Excellent	None	81	No lifting	Slight cracking	Fail–2.3 (20) Dir & Fail–3.4 (30) Indir
		9	Very good	None	75	Slight lifting along scribes	Pass	Fail–3.4 (30) Dir & Fail–2.3 (20) Indir
		12	Very good	None	68	Slight lifting along scribed	Pass	Fail–6.8 (60) Dir & Fail–8.5 (75) Indir

*Unexposed (control)

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(continued)

TABLE 19. EFFECT OF OUTDOOR WEATHERING ON PERFORMANCE OF PROTECTIVE COATING SYSTEMS (Continued)

Coating System	Substrate	Exposure Time, Months	Performance Evaluation Factors					
			Appearance	Corrosion	Gloss	Adhesion	Flexibility	Impact Resistance, J (in.-lb)
Polyamide Power Spray	Steel	0*	Good	–	30	Pass	Pass	Pass–8.5 (75) Dir & Indir
		3	Color shift	Some on edges	30	Lifting along scribes	Cracked & peeled from substrate	Fail–4.5 (40) Dir & Fail–1.1 (10) Indir
		6	Good	Very heavy on edges	24	Lifting along scribes	Cracked & peeled from substrate	Fail–3.4 (30) Dir & Fail–1.1 (10) Indir
		9	Almost no paint left	Very severe	Not tested	Not tested	Not tested	Not tested
		12	Almost no paint left	Very severe	Not tested	Not tested	Not tested	Not tested

*Unexposed (control)

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SECTION 6

REPLACEMENT COATINGS FOR CYANIDE COPPER PLATING

APPROACH

This phase of the program was directed toward evaluating potential replacement systems for cyanide-type copper electroplating. The approach taken involved evaluating five non-cyanide copper electroplating systems and comparing the results to a cyanide-type control system.

STUDY AREAS

Evaluation of the non-cyanide copper plating systems involved the following:

- Selection of substrates, pretreatment procedures, and application procedures
- Testing and evaluation of coatings
- Economic and environmental evaluation.

BACKGROUND

Copper is normally plated from cyanide-type electroplating baths. However, the high level of toxicity of the cyanides necessitates the use of expensive waste treatment procedures involving either chlorine or hypochlorites to destroy the cyanides when these solutions are dumped. Several cyanide-free copper electroplating baths are available and have potential as replacement systems. The following cyanide-free systems were evaluated and compared to Mac Dermid's Rocheltex cyanide copper plating system:

- Cu-Pure - Lea-Ronal, Inc.
- Enthobrite Cu-942 - Enthone, Inc.
- Copper Fluoborate - Harstan, Inc.
- Ac-94 Bright Acid Copper - M&T Chemicals, Inc.
- Unichrome Pyrophosphate - M&T Chemicals, Inc.

These systems include two alkaline types (Cu-Pure and Unichrome Pyrophosphate), two acid sulfate types (Cu-942 and Ac-94), and one acid fluoborate type, (Copper Fluoborate).

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SUBSTRATE PRETREATMENT AND COATING APPLICATION

Substrates to which copper plate is commonly applied include 4340 and 300M high-strength steels. High-strength 4340 steel was chosen as the principal substrate for this study. Pretreatment of the substrate is similar to that used for cyanide copper plating, i.e., solvent clean, alkaline clean, and vapor blast. A strike is required to provide proper adhesion of the copper plate. In general, a strike is required for all copper plating baths, cyanide and non-cyanide, except the Rochelle-type cyanide baths.

Each of the plating baths was prepared according to manufacturers' recommendations. Elevated-temperature additions and carbon treatment were used in the bath makeup, as required. The baths were maintained and operated as suggested by the manufacturers (Table 20). All but one of the non-cyanide baths evaluated (Lea-Ronal's Cu-Pure) was operated at ambient temperature. In general, the cyanide-type baths (except those used for striking) must be operated at elevated temperatures (over 60 °C (140 °F)).

Standard laboratory equipment was used for the Hull cell tests and the 2.51 (0.66 gal) solution tests. Zinc-plated steel Hull cell panels were stripped of zinc in 50% HC1, rinsed, dipped in 20% H_2SO_4 to prevent chloride drag-in, and rinsed again prior to striking in a Watts nickel bath and copper plating in a 267 ml (0.07 gal) Hull cell.

TESTING AND EVALUATION OF NON-CYANIDE COPPER PLATING BATHS

The five non-cyanide copper plating systems were screened in Hull cell tests to determine the bright range and throwing power of the baths (Table 21). Hull cell panels were nickel-struck and plated at 3 A for 3 min in a 267 ml (0.07 gal) Hull cell. As indicated in Table 21, Enthobrite Cu-942 had the greatest bright range (11-1290 A/m^2 or 1-120 A/ft^2) with excellent throwing power. Leo-Ronal's Cu-Pure and M&T's AC-94 had a bright range and throwing power equivalent to that of the cyanide-type control. The throwing power of Harstan's Fluoborate and M&T's Pyrophosphate was not as good as that of the other systems, although the bright ranges were similar to those of the other systems. Cu-Pure and Enthobrite Cu 942 were selected for further testing because they have the best combination of cost, maintenance requirements, and bright range and throwing power of all the systems evaluated.

Final screening of Cu-Pure and Cu-942 was performed on panels plated in a 2.51 (0.66 gal) plating bath. Steel panels were nickel-struck and plated for 40 min at the optimum current density for that system, as determined by the Hull cell tests. While gassing was observed during plating from the cyanide-type control system and foaming was observed during plating from Lea-Ronal's Cu-Pure solution, no gassing or foaming was observed during plating from Enthone's Cu-942 solution. Enthone's Cu-942 produced a smooth and bright plate with no edge burning, while the cyanide-type control and Lea-Ronal's Cu-Pure showed some edge-burning and lacked brightness. The plating rate for Cu-942 was also 60 to 100% higher than the cyanide-type control and Lea-Ronal's Cu-Pure. In addition, Enthone's Cu-942 provided better protection against decarburization of steel subjected to heat treatment (830 °C (1525 °F) for 10 min) than either the cyanide-type control or Lea Ronal's Cu-Pure. Metallographic examination of panels subjected to heat treatment was performed to determine the protection offered the steel from decarburization by the copper coating. Panels of 4130 steel (2.54 x 101.6 x 1.02 mm (1 x 4 x 0.040 in.)) were copper-plated for 40 min at the optimum current density of the system and subjected to a full heat-treat cycle of 830 °C (1525 °F) for 10 min. After the heat-treat cycle, none of the copper plates (cyanide control, Cu-Pure or Cu-942) showed evidence of blistering or cracking. The panels were stripped in Enstrip A. The panels protected with the cyanide copper plate showed some carbon deposits and some evidence of decarburization in the metallographic study. Minute decarburization was evident in the metallographic examination of the panels protected with Cu-Pure. There were no noticeable carbon deposits on these panels. The metallographic study of the

TABLE 20. NON-CYANIDE COPPER PLATING SYSTEMS - OPERATING CONDITIONS

Plating system	Component	Concentration
MacDermid-Rocheltex (cyanide type control)	CuCN	56.3 g/l (7.5 oz/gal)
	KCN	105 g/l (14.0 oz/gal)
	KOH	18.8 g/l (2.5 oz/gal)
	Rocheltex	7% by volume
	S-1 brightener	1% by volume
	Temperature	63 ± 2.5°C (145 ± 5°F)
Lea Ronal - Cu Pure	Cu-pure concentrate	33.33% by volume
	KOH	17.0% by volume
	Cu-pure H501	1.0% by volume
	Cu-pure L301	0.125-0.25% by volume
	Cu-pure wetting agent	0.25% by volume
	Temperature	60-70°C (140-160°F)
Enthone-Enthobrite Cu-942	$CuSO_4$	210 g/l (28 oz/gal)
	H_2SO_4 66° Be	31.5 g/l (4.2 oz/gal)
	HCl 20° Be	11.3 g/100 l (1.5 oz/100 gal)
	Cu 942 M	480 g/100 l (64 oz/100 gal)
	Cu 942 R	97.5 g/100 l (13 oz/100 gal)
	Cu 942 S	6 g/100 l (0.8 oz/100 gal) (max)
Harstan - copper fluoborate	Copper fluoborate	351 g/l (46.8 oz/gal)
	Copper metal	93.8 g/l (12.5 oz/gal)
	Fluoboric acid	22.5 g/l (3.0 oz/gal)
	Boric acid	22.5 g/l (3.0 oz/gal)
	Shinol C-F #1	9.8 g/l (1.3 oz/gal)
M&T - Ac94 bright acid copper	$CuSO_4$	225 g/l (30 oz/gal)
	H_2SO_4 66° Be	30 g/l (4 oz/gal)
	Chloride ion	60 ppm
	Ac-94 makeup	0.4% by volume
	Ac-94 leveler	0.1% by volume
M&T - unichrome pyrophosphate	Copper metal	22.5 g/l (3 oz/gal)
	P_2O_7: Cu ratio	7.7:1
	NH_3	1.2 g/l (0.16 oz/gal)
	NH_4OH 26° Be	4.65 g/l (0.62 oz/gal)

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TABLE 21. NON-CYANIDE COPPER PLATING - SCREENING TESTS

	Hull cell tests		2.5-liter (0.66-gal) solution tests						
System	**Bright range, A/m^2 (A/ft^2)**	**Throwing power**	**Current density, A/m^2 (A/ft^2)***	**Surface condition**	**Edges**	**Comments**	**Plating rate, μm/min (mil/min)**	**RMS value**	**Heat treat evaluation**
Macdermid Rocheltex (cyanide-type control)	11-387 (1-36)	Good	270 (25)	Smooth	Slight burning	Excessive gassing	0.495 (0.020)	55-100	Some decarb
Lea-Ronal Cu-Pure	11-324 (1-30)	Good	390 (36)	Smooth	Some burning	Some foaming	0.406 (0.016)	60-125	Minute decarb
Enthone Cu-942	11-1290 (1-120)	Excellent	780 (72)	Smooth & bright	No burning	No gassing or foaming	0.813 (0.032)	45-55	No decarb
Harstan Fluoborate	11-774 (1-72)	Poor							
M&T AC-94	11-324 (1-30)	Good							
M&T Pyrophosphate	11-234 (1-22)	Poor							

*Optimum current density as determined by Hull cell tests

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Cu-942-protected panels showed no evidence of decarburization. The steel was clean and free from any carbon deposits. Enthone's Cu-942 was selected as the best candidate for further testing based on these screening test results.

In order to obtain good adhesion of the copper plate, a strike was necessary. A Watts nickel bath (Table 22) was used with good results. Samples were struck for 1 min in the nickel bath at an optimum current density of 430 A/m^2 (40 A/ft^2). Although the nickel strike tends to passivate quickly, it can be activated by a 1-min dip in dilute H_2SO_4 immediately prior to copper plating. Adhesion and solderability tests show the affect of passivation/reactivation of the nickel strike on the adhesion of the copper plate (Table 23). Although the adhesion and solderability of the copper plate on a reactivated nickel strike is satisfactory, copper plating immediately following the nickel strike produces superior results and is preferred. Testing showed that optimum plating results are obtained under the operating conditions recommended by the supplier (Table 24). The optimum operating temperature is 21 to 27°C (70 to 80°F); at higher temperatures, loss of leveling and brightness occurs.

TABLE 22. WATTS NICKEL BATH COMPOSITION

Solution	Concentration
$Ni_2\ SO_4$	278.3-315.9 g/l (2.32-2.64 lb/gal)
NiCl	45.2-75.2 g/l (0.38-0.63 lb/gal)
H_3BO_3	22.6-52.7 g/l (0.19-0.44 lb/gal)
$H_2\ O_2$ (30-35%)	0.5-1.5% by volume
p^H	1.5-4.5

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TABLE 23. EFFECT OF NICKEL STRIKE ON ENTHOBRITE Cu-942 NON-CYANIDE COPPER PLATING

Condition	Adhesion (bend test)	Solderability (bend test)
No nickel strike prior to copper plating	Poor (no adhesion)	–
Nickel strike passivated; reactivated w/H_2SO_4 immediately prior to copper plating	Good (minor lifting)	Fair (some lifting)
Nickel strike immediately prior to copper plating	Excellent (no lifting)	Excellent (no lifting)

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TABLE 24. ENTHOBRITE Cu-942 COPPER PLATING - OPERATING CONDITIONS

Operating conditions	Range	Optimum
Copper sulfate, g/l	203 - 225	210
(oz/gal)	(27 - 30)	(28)
Sulfuric acid, ml/l	27.3 - 37.5	32.8
(fl oz/gal)	(3.5 - 4.8)	(4.2)
Hydrochloric acid, ml/1000 l	7.8 - 15.6	11.7
(fl oz/100 gal)	(1 - 2)	(1.5)
Cathode current density,	54 - 1076	646 - 753
A/m^2 (A/ft^2)	(5 - 100)	(60 - 70)

Sustained load tests were run to determine the extent of hydrogen absorption in acid copper-plated material and to evaluate the potential for embrittlement failure. Nine sustained-load notched tensile specimens, configured as shown in Figure 9, of 4340 steel heat-treated to 1790-1930 MPa (260-280 ksi) were tempered at 650 °C (1200 °F) for 2 hr to return them to a low strength level typical of production work submitted for copper plating. Six specimens were plated, three in cyanide copper and three in acid copper, and treated as follows:

- Plate to 76-102 μm (3-4 mils)
  - 3 cyanide copper specimens (controls)
  - 3 acid copper specimens (Cu-942)
- Heat treat to 1790-1930 MPa (260-280 ksi)
- Strip in Enstrip A.

Three specimens were acid copper plated and treated as follows:

- Plate to 51-76 μm (2-3 mils) - three acid copper specimens (Cu-942)
- Normalize at 870 °C (1600 °F)
- Strip in Enstrip A
- Replate to 25-51 μm (1-2 mils)
- Heat treat to 1790-1930 MPa (260-280 ksi)
- Strip in Enstrip A.

All of the specimens were then loaded at 75% of the ultimate notched tensile strength (1860 MPa (270 ksi) for over 200 hr without failure.

The characterization of Enthobrite Cu-942 showed that this non-cyanide copper plating system exceeded the requirements established for copper plating under MIL-C-14450 (Table 25). The adhesion and solderability of the copper plate were excellent. No hydrogen embrittlement was evident in notched tensile tests of the Cu-942-plated specimens. The Cu-942 plating adequately protected the steel from decarburization during heat treatment. Several of the copper-plated test panels are shown in Fig. 26.

ECONOMIC AND ENVIRONMENTAL EVALUATION

Economic evaluation of the electroplating operation includes consideration of the equipment requirements, plating chemicals, bath maintenance, and waste treatment requirements. Toxic chemicals used, discharge levels, safety precautions, and waste treatment requirements are included in the environmental evaluation.

The equipment used for non-cyanide copper plating is similar to that used for cyanide copper plating. Because of the corrosive nature of acid copper sulfate solutions, equipment must be made of acid-resistant materials. Plating tanks of polypropylene, polyethylene-lined fiberglass, or PVC-lined steel are recommended. Other equipment that may come in contact with the solution, such as floors, pipes, and tank exteriors, should be coated with acid-resistant paint. As is the case with cyanide copper plating, air agitation and continuous filtration are necessary to provide optimum plating results. In addition, cooling must be provided to maintain the bath at 21-27 °C (70-80 °F). Installation costs for new plating equipment will typically be the same for cyanide and non-cyanide copper plating systems. Change-over costs from existing cyanide plating equipment to a non-cyanide plating system will be minimal, involving only the cost of lining tanks.

Table 25. Enthobrite Cu-942 Non-Cyanide Copper Plating Characterization Tests

Property	Procedure	Results
Thickness	Permascope	28-33 μm @ 0.813 μm/min (1.1-1.3 mils @ 0.032 mils/min)
Adhesion	Sheet Bend	Excellent*
Decarburization protection	Metallographic examination	No decarburization
Solderability	Solder 232°C (450°F)-sheet bend	Excellent*
Hydrogen embrittlement	75% unts/200 hr	Pass

* Copper plate immediately following nickel strike

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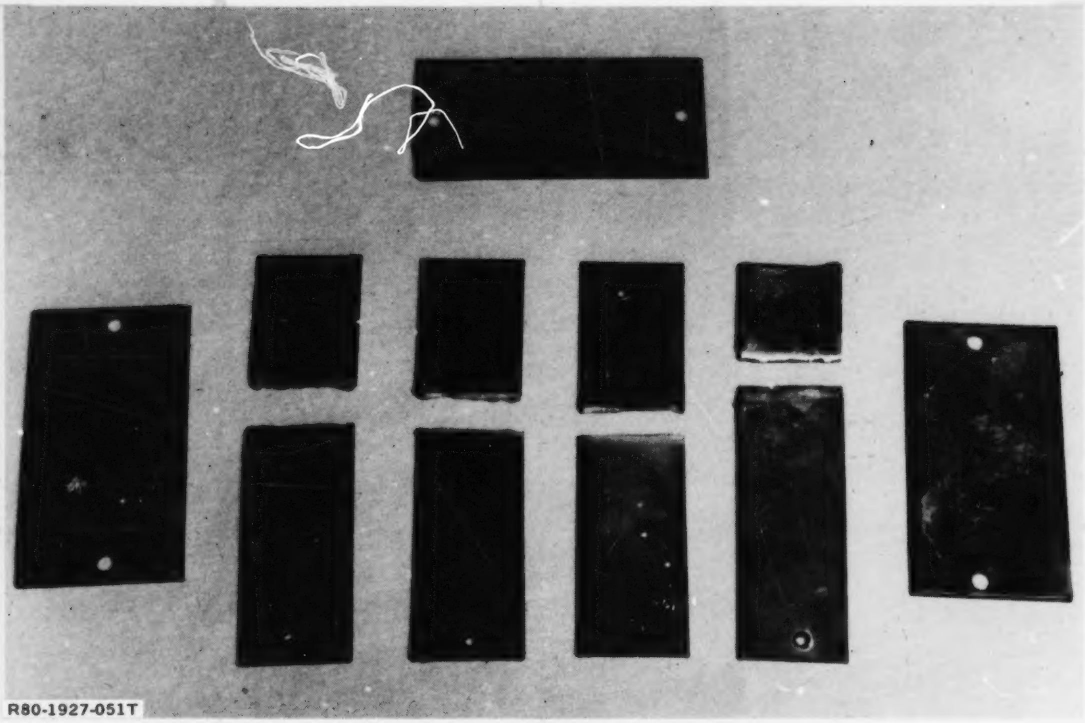

Figure 26. Non-Cyanide Copper-Plated Tests Panels: Enthone's Enthobrite Cu-942.

Cost for pretreatment and the associated equipment will also be the same for both cyanide and non-cyanide copper plating. Pretreatment of the substrate prior to plating is the same with both types of plating baths. As is the case with all electroplating operations, thorough cleaning of the parts is necessary to ensure maximum adhesion of the plate and to minimize contamination of the bath. Steel substrates are pretreated by solvent cleaning, alkaline cleaning and vapor blasting, or by other, similarly effective pretreatment techniques. A strike is required on steel to provide proper adhesion of the copper plate for both cyanide and non-cyanide type copper plating, except for the Rochelle-type cyanide copper plating which provides adequate adhesion without a strike. Since a cyanide copper strike or an acid nickel strike can be used for the acid copper plating system, it is possible to completely eliminate cyanides from the plating system.

The plating chemicals required for Enthobrite Cu-942 and Mac Dermid Rocheltex cyanide copper plating baths are shown in Table 26. Comparison of the chemical costs for make-up of 378 1 (100 gal) shows that the cost of the non-cyanide copper plating bath is approximately one-third that of the cyanide copper plating bath. Although the cost difference is not as dramatic for all copper plating system comparisons, it does show the potential for substantial cost savings in bath makeup costs. Maintenance of the copper plating bath requires regular additions of several proprietary chemicals to ensure optimum plating results. An ampere-hour meter and other supplementary control meters are used to aid in the control of both cyanide and non-cyanide copper plating baths.

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TABLE 26. COPPER PLATING - MAKEUP COST COMPARISON

Non–Cyanide Copper (Enthobrite Cu-942)				Cyanide Copper (MacDermid Rocheltex)			
Chemical	Qty/100 gal	Cost, $/gal	Total Cost, $/100 gal	Chemical	Qty/100 gal	Cost, $/gal	Total Cost, $/100 gal
$CuSO_4$	21.88 gal	2.96 ***	64.66	CuCN	11.72 gal	7.76 **	90.90
H_2SO_4 66°Be	3.28 gal	0.44 *	1.44	KCN	21.87 gal	2.68 **	58.63
HCl 20°Be	1.5 oz	5.97	0.07	KOH	3.91 gal	1.85 **	7.25
Cu 942 M	0.50 gal	8.30	4.15	Rocheltex	7.00 gal	6.90	48.30
Cu 942 R	0.10 gal	8.40	0.84	S-1 Brightener	1.00 gal	6.25	6.25
Cu 942 S	0.8 oz	8.00	0.05	Total			211.33
Total			71.21				

* 1974 PRICES ** 1975 PRICES ***1976 PRICES

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Cyanide copper plating systems require extensive waste treatment of the spent electroplating solution to remove the cyanides. This waste treatment includes either the destruction of cyanides through the chlorination process or by reaction with hypochlorites, or by conversion to cyanate using chlorine, hypochlorites, or hydrogen peroxide. Each of these methods introduces a chemical waste treatment step of substantial cost into plating operations. Non-cyanide copper plating systems do not require this costly waste treatment step. These systems require only the precipitation of the copper by adjusting the pH to 8.5 to 9.0. In addition to substantially reducing the waste treatment costs by eliminating the cyanides from the plating process, worker safety is greatly increased.

SECTION 7

REPLACEMENT COATINGS FOR HEXAVALENT CHROMIUM PLATING

APPROACH

This phase of the program was directed toward evaluating a potential replacement system for hexavalent chromium plating. The approach taken involved the evaluation of a trivalent chromium electroplating process and comparison to a hexavalent chromium system as a control.

STUDY AREAS

Evaluation of the trivalent chromium plating system involved the following:

- Selection of substrates, pretreatment procedures, and application procedures
- Testing and evaluation of coatings
- Economic and environmental evaluation.

BACKGROUND

Chromium is normally plated from hexavalent chromic acid electroplating baths for decorative applications such as home appliances, marine hardware, automobile hardware, zinc die casting, brass forgings, and steel stampings. However, the toxicity and waste treatment requirements of hexavalent chromium compared to those of trivalent chromium make a trivalent chromium plating system desirable.

The maximum permissible concentration of hexavalent chromium for public water supplies is 0.05 mg/1 (0.00000066 oz/gal). The hexavalent chromium must be reduced with sulfur dioxide or sodium metabisulfite to trivalent chromium before it can be precipitated as insoluble chromium hydroxide from spent electroplating baths. Chromium reduction is a costly step in the the waste treatment process required to meet discharge standards.

In addition, a toxic spray is produced during operation of the hexavalent chromic acid baths. This can be detrimental to the workers health, since chromic acid and its salts have a corrosive action on the skin and mucous membranes. The characteristic lesion is a deep penetrating ulcer which, for the most part, does not tend to suppurate and which is slow in healing.

Use of a trivalent chromium plating bath would eliminate both of these problems. Trichrome, the trivalent chromium plating process developed by Albright and Wilson, Oldbury, England, and marketed by Harshaw Co., Cleveland, Ohio, was evaluated in comparison to a standard hexavalent chromic acid system. Trichrome is a decorative chromium plating system. The trivalent chromium process has not yet been extended to hard chromium.

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SUBSTRATE PRETREATMENT AND COATING APPLICATION

Substrates to which chromium plating is commonly applied include 1010-1020 steel and 4340 high-strength steel. The pretreatment procedure includes vapor degreasing, alkaline cleaning, and reverse-etching of the metal surface to be plated. A nickel underplate is required for decorative chromium plating to provide good plate adhesion and maximum corrosion protection. Panels were sent to Harshaw Chemical Company for coating application, which was done according to their recommended procedure. The chromium was applied over a duplex nickel (25.4 μm (1.0 mil) semi-bright nickel, 10.2 μm (0.4 mil) bright nickel) to a thickness of 0.25 μm (0.01 mil). The bath operating conditions are shown in Table 27. The bath is operated at ambient temperature.

TABLE 27. TRICHROME - OPERATING CONDITIONS

Component	**Optimum concentration**
TC additive	390 g/l (52 oz/gal)
TC stabilizer	80 ml/l (8% by vol)
TC - SA	12 ml/l (1.2% by vol)
TC regulator	1.0 ml/l (4 qt/1000 gal)
Total chromium	20 g/l (2.7 oz/gal)
Parameter	**Optimum condition**
Temperature	23°C (72°)
pH	3.2
Current density:	
Cathode	970 A/m^2 (90 A/ft^2)
Anode	540 A/m^2 (50 A/ft^2)
Anodes	Harshaw special graphite
Agitation	Mild air agitation
Deposition rate	0.075-0.125 μm/min (0.003-0.005 mil/min) @ 1080 A/m^2 (100 A/ft^2)

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TESTING AND EVALUATION OF TRIVALENT CHROMIUM

Panels of 1010 steel were cleaned and plated with 25.4 μm (1.0 mil) semi-bright nickel, 10.2 μm (0.4 mil) bright nickel, and 0.25 μm (0.01 mil) chromium. The coating performance was screened with respect to appearance, corrosion, and adhesion. Initially, the coating was applied to rough panels, as received from stock. Due to unsatisfactory results obtained with these panels (Table 28), additional panels were buffed to a smooth finish characteristic of typical plated parts prior to cleaning and plating.

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TABLE 28. TRIVALENT CHROMIUM PLATING: EVALUATION OF PROPERTIES

Property	Trichrome* (unbuffed 1010 steel)	Trichrome* (buffed 1010 steel)	Hexavalent chromium* (buffed 1010 steel)
Appearance	Bright	Smooth & very bright	Smooth & bright
Adhesion (bend test)	Poor	Good	Good
Corrosion (salt spray)	96 hr**	282 hr***	96 hr**

* 25.4 μm (1.0 mil) semi-bright nickel
10.2 μm (0.4 mil) bright nickel
0.25 μm (0.01 mil) chrome (trivalent or hexavalent)

** Each of 4 panels failed at 96 hr

*** Average of 4 panels; 2 failed at 48 hr, 1 failed at 288 hr, 1 failed at 744 hr

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Results of the screening tests on the buffed panels showed that the trivalent chromium produced excellent results in comparison to the hexavalent chromium (Ref. Table 28). The appearance of both the trivalent and hexavalent chromium plate was excellent. Both types were smooth and bright, with the trivalent plate being somewhat brighter than the hexavalent plate. By comparison, the unbuffed panel plated with Trichrome was bright but not smooth, magnifying the orange-peel effects of the unbuffed panels. Plate adhesion was evaluated by bending the panels to break and examining the break at 4X magnification for lifting or peeling. No separation was evident at the chromium-nickel interface, the nickel-nickel interface, or the nickel-base metal interface on either the Trichrome or hexavalent chromium plates on the buffed panels. The adhesion of the plate to the unbuffed panel, on the other hand, was poor (the underplate lifted from the substrate and the chromium separated from the underplate). This loss of adhesion was apparently due to the roughness of the substrate. The corrosion resistance was determined by exposure of four plated panels to 5% salt spray solution until failure. Two trichrome buffed panels failed at 48 hr, one at 288 hr, and one at 744 hr (an average of 282 hr). This average is considerably higher than that obtained with the hexavalent chromium panels, all four of which failed at 96 hr. Although the variation in the Trichrome results indicate that this bath may need further work to provide consistently higher corrosion protection, the results do show that Trichrome has excellent potential for providing improved corrosion protection over the hexavalent chromium while maintaining the performance in other properties. Several of the plated test panels are shown in Fig. 27 (trivalent chromium) and Fig. 28 (hexavalent chromium).

Hydrogen embrittlement tests were performed in addition to the above tests to provide final characterization of the chromium plating systems. Six notched tensile specimens of 4340 steel, configured as shown in Fig. 9, were heat treated to 1790-1930 MPa (260-280 ksi) and plated, as described above, with duplex nickel and chromium. Three specimens were plated with Trichrome and three specimens were plated with hexavalent chromium. All specimens were baked for 3 hr at 191 °C (375 °F) following plating to provide embrittlement relief. Three bare control specimens were installed in a universal testing machine and continuously loaded until failure occurred to determine the ultimate notched tensile strength (UNTS). The average UNTS was found to be 56.5 MPa (389.3 ksi). The plated specimens were then subjected to a sustained load of 75% of the ultimate notched tensile strength

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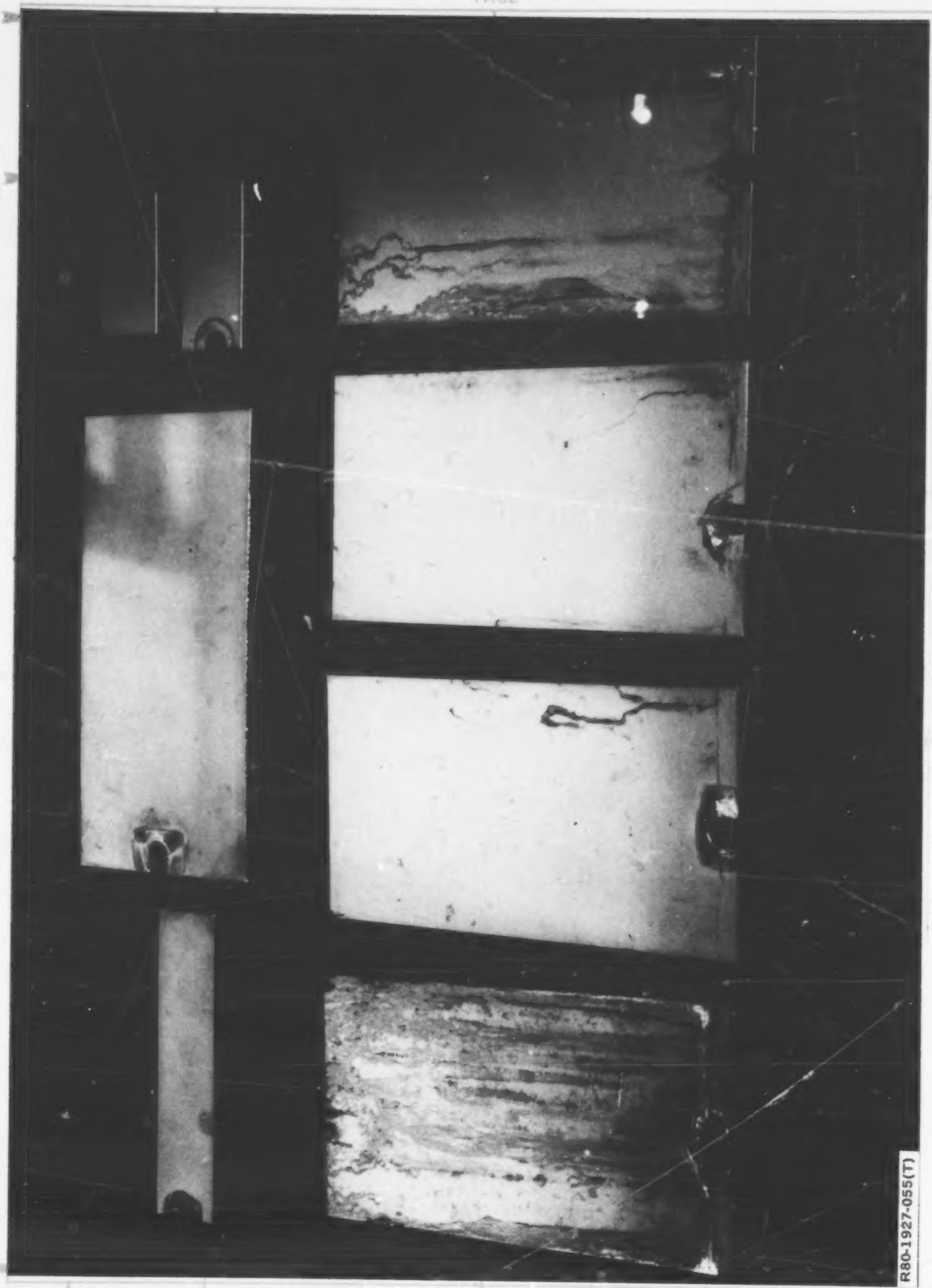

Figure 27. Trivalent Chromium-Plated Test Panels.

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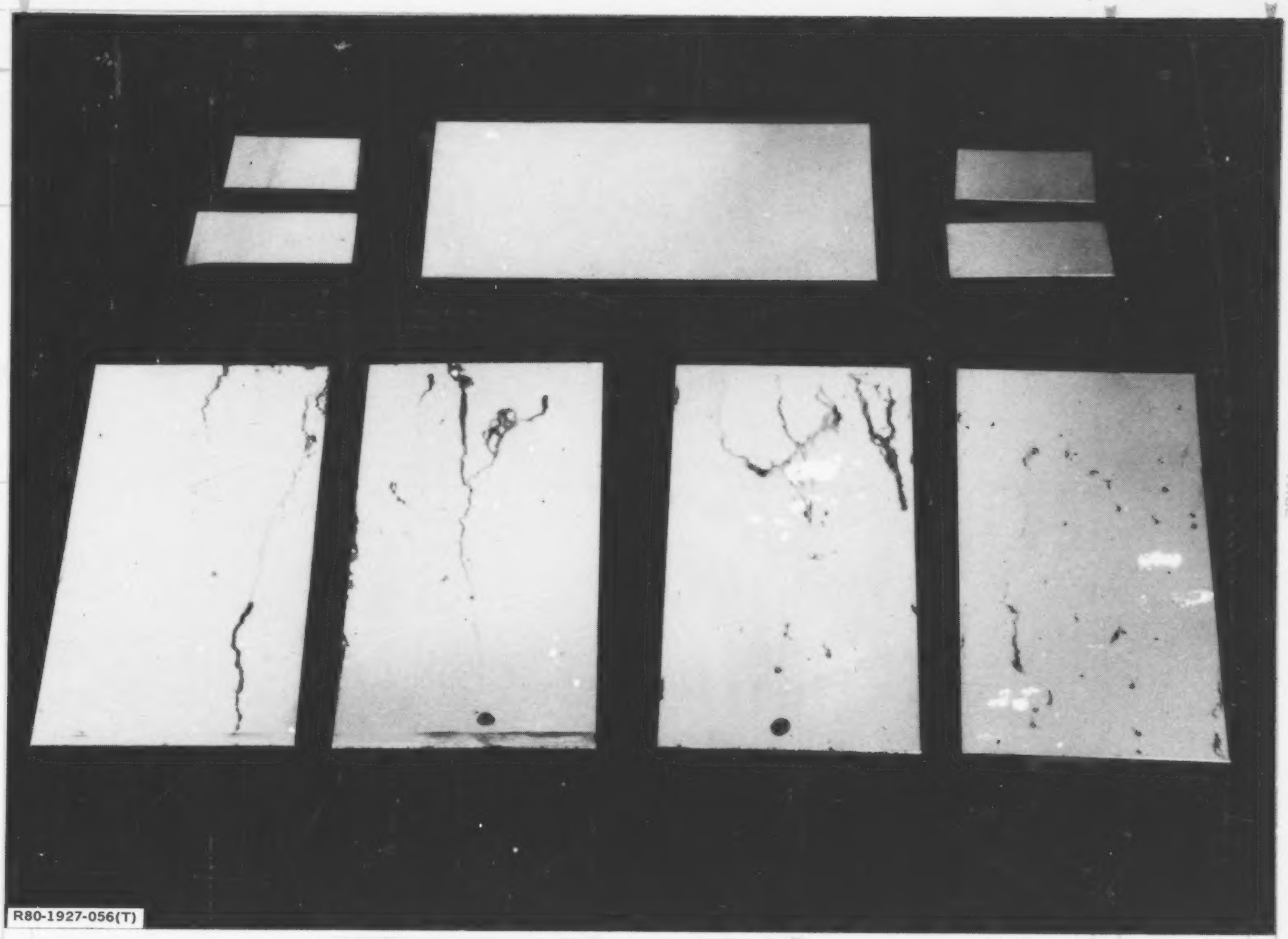

Figure 28. Hexavalent Chromium-Plated Test Panels.

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for 200 hr. The Trichrome-plated specimens exceeded the 200-hr exposure with no failure (Table 29). The hexavalent chromium-plated specimens all failed in less than 7 hr. Since chromium plating on high-strength steel is normally baked for 23 hr to provide complete hydrogen embrittlement relief, hexavalent chromium should pass the notched tensile test. Differences in the nickel baths used for the trivalent and hexavalent chromium specimens may also have contributed to these failures.

TABLE 29. NOTCHED-TENSION SUSTAINED-LOAD TESTS: CHROMIUM PLATING

Coating system	Substrate	Load (0.75 x ult*), kn (lb)	Spec no.	Test duration,** hr	Remarks
Hexavalent chromium plated	4340	31.2 (7020)	9	5.2 fail	Baked 3 hr @ 191°C (375°F)
			11	6.1 fail	
			29	3.3 fail	
Trivalent chromium plated (Trichrome)	4340	31.2 (7020)	24	260 NF ‡	Baked 3 hr @ 191°C (375°F)
			32	260 NF	
			34	260 NF	

*Ultimate load:

Substrate	Spec. no.	Ult. load, kn (lb)	Notch ten. str., MPa (ksi)
4340	14	41.5 (9330)	56.3 (388.1)
	15	41.7 (9365)	56.5 (389.5)
	28	41.7 (9380)	56.6 (390.2)
	avg	41.6 (9358)	56.5 (389.3)

**Minimum requirement: 200 hr @ 75% of ultimate notch tensile strength

‡ NF - no failure

# All specimens plated with 25.4 μm (1.0 mil) semi-bright nickel
10.2 μm (0.4 mil) bright nickel
0.25 μm (0.01 mil) chromium (trivalent or hexavalent)

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ECONOMIC AND ENVIRONMENTAL EVALUATION

In the replacement of hexavalent chromium with trivalent chromium, several economic considerations must be made. The cost of converting hexavalent chromium equipment for trivalent chromium service, or the difference between new installation costs, should be considered. The difference in cost of pretreatment plating chemicals, bath maintenance, and waste treatment should also be considered. The effect on the environment and the health and safety of workers are other important considerations.

The main equipment consideration in the conversion of chromium plating is the electroplating tanks. The plating tanks used for trivalent chromium plating must be plastic or rubber. Lead-lined hexavalent chromium plating tanks must be converted by lining with 6.4 mm (0.25 in.) rigid plastic sheet before use with a trivalent chromium bath, since lead is a contaminant to this bath. Special carbon anodes fitted with titanium anode hooks are used for the trivalent chromium process. These anodes do not deteriorate or need replacing as the lead anodes used for the hexavalent chromium process do. Air agitation and cooling must be supplied for the plating tank. The filter must be resistant to high-chloride-content solutions. The same 6-9 volt rectifiers can be used for both types of chromium plating.

Of the other cost items that need to be considered, the pretreatment and underplating requirements are the same. The bath makeup costs for Trichrome are $0.90/1 ($3.40/gal); costs for a typical hexavalent chromium bath are about $0.53/1 ($2.00/gal). Bath maintenance costs are also reported to be aout 20% higher for the trivalent bath. Waste treatment costs for the trivalent chromium are estimated to be $0.02/1 ($0.07/gal) compared to $0.15/1 ($0.55/gal) for hexavalent chromium.

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SECTION 8

REPLACEMENT SYSTEMS FOR PHENOLIC PAINT STRIPPERS

APPROACH

This phase of the program was directed toward evaluation of potential replacement systems for phenolic paint strippers. The approach taken involved the evaluation of several non-phenolic paint strippers of the acid and non-acid types for brush and immersion applications, and selection of the best system of each type.

STUDY AREAS

Evaluation of the replacement systems for non-phenolic paint stripper systems involved the following:

- Selection of substrates, test coatings, and stripping procedures
- Testing and evaluation of candidate paint strippers
- Economic and environmental evaluation.

BACKGROUND

Paint strippers are widely used throughout the metal-finishing industry to remove paint from parts to permit repair, inspection, or refinishing. Most paint strippers contain chromates, methylene chloride, phenols, or strong acids. The normal procedure involves application of the paint stripper, allowing it to remain on the part for a specified period and then washing it off with water. In many cases, the wash water containing the paint stripper and removed paint is fed into drains that go to leaching ponds or sewers. This can result in serious water-pollution, especially when highly toxic chromates or phenols are involved.

Several paint strippers containing no phenols have been developed as potential replacements for phenolic paint strippers. The systems that were considered for potential replacement of phenolic paint strippers are shown in Table 30.

SUBSTRATE COATING AND STRIPPING PROCEDURES

The coating used for all paint stripper tests was the MIL-P-23377 epoxy primer/MIL-C-81773 polyurethane topcoat system. This coating was applied to 2024-T3 aluminum alloy panels according to conventional procedures and air-dried for 7 days prior to use in paint stripper evaluations. The paint surface to be stripped was completely covered with remover, either by immersion or by brush. After the required contact time, the area was rinsed with water and the loosened paint removed by brushing.

TABLE 30. NON-PHENOLIC PAINT STRIPPER SYSTEMS EVALUATED

Manufacturer	Trade name	Application	Acid/non-acid
BASF	Sprazee	Brush on	Non-acid
BASF	Rapoff	Immersion	Non-acid
Magnus	MB-7506	Brush on	Non-acid
Magnus	766	Immersion	Non-acid
Oakite	Stripper 157	Brush on	Non-acid
Oakite	Stripper EZ	Immersion	Acid
Oakite	Stripper SA	Immersion	Acid
Oakite	Visstrip	Brush on	Acid
Randolph	Randostrip	Brush on	Non-acid
Turco	T-2822, thick	Brush on	Acid
Turco	T-5873	Brush on	Non-acid
Turco	T-6017	Brush on	Acid
Turco	T-6215	Brush on	Non-acid
Turco*	T-5469	Brush on	Phenolic/non-acid

*Tested as phenolic control system

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TESTING AND EVALUATION OF REPLACEMENT SYSTEMS

Testing of potential replacement systems for phenolic paint strippers was performed to determine the best system available of each of the four types: acid brush-on, non-acid brush-on, acid immersion, and non-acid immersion. Strippers in each category were subjected to various screening tests applicable to that category and compared to the phenolic non-acid brush-on type stripper used as a control. Preliminary screening tests were conducted to determine the stripper's remover power, rinsability, coating and remover residue, and general properties. The coating system used in these tests was an epoxy primer conforming to MIL-P-23377 with a polyurethane topcoat conforming to MIL-C-81773 on an alodined 2024-T3 clad aluminum substrate.

Preliminary screening tests results for the brush-on application strippers are shown in Table 31. The performance of the six candidate non-acid brush-on strippers varied widely; the removal power, or time required for completion of lifting, ranged from 20 to more than 80 min. This time was determined as that required for completion of all lifting and wrinkling action, using a remover volume sufficient

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TABLE 31. BRUSH-ON PAINT STRIPPERS: PRELIMINARY EVALUATION

Manufacturer	Trade Name	Appearance	Consistency & Flow	Removal Power (time to lift, min:sec)	Rinsability	Coating Residue (% Removed)	Remover Residue
Phenolic control							
• Turco	T-5469	Homogeneous	Smooth, even coat	5:17	Excellent	> 99%	Much residue
Non-phenolic, non acid							
• BASF	Sprazee	Homogeneous	Fairly even coat	56:50	Good	> 98%	Easily rinsed
• Magnus	MB-7506	Slight separation	Smooth, even coat	> 80:00	Fair	60% Req brushing	Some residue
• Oakite	Stripper 157	Homogeneous	Fairly even coat	> 80:00	Fair	95% Req brushing	Much residue
• Randolph	Rando-strip	Separated	Smooth, even coat	46:46	Fair	25% Req brushing	Some residue
• Turco	T-5873	Homogensous	Smooth, even coat	20:05	Excellent	> 99%	Much residue
• Turco	T-6215	Homogeneous	Fairly even coat	> 80:00	Good	99% Req brushing	Easily rinsed
Non-phenolic, acid							
• Oakite	Visstrip	Separated	Smooth, even coat	8:28	Excellent	95%	Easily rinsed
• Turco	T-2822	Homogeneous	Smooth, even coat	8:18	Excellent	> 99%	Easily rinsed
• Turco	T-6017	Slight separation	Fairly even coat	3:20	Excellent	> 99%	Easily rinsed

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TABLE 32. BRUSH-ON PAINT STRIPPERS: CHARACTERIZATION TESTS

Manufacturer	Trade name	Appearance	Consistency & flow	Remover power, (time to lift, min:sec)	Remover power			Rinsability	Coating residue (% removed)	Remover residue	Refinishing properties		Corrosion compatibility weight change, %**	
					Contact time (min: sec)	Remover 10 ml (% lifted)	Volume 25 ml (% lifted)				Gloss*	Adhesion	Aluminum	Steel
Phenolic control														
• Turco	T-5469	Homogeneous	Smooth, even coat	5:17	5:30	100%	100%	Excellent	>99%	Much residue	91	Good	-0.011%	+0.007%
Non-phenolic, non-acid														
• BASF	Sprazee	Homogeneous	Fairly even coat	56:50	30:00	82%	97%	Good	98%	Easily rinsed	N.D.	N.D.***	N.D.***	N.D.***
• Turco	T-5873	Homogeneous	Smooth, even coat	20:05	6:45	42%	100%	Excellent	>99%	Much residue	90	Good	+0.007%	+0.031%
Non-phenolic, acid														
• Oakite	Visstrip	Separated	Smooth, even coat	8:28	5:20	92%	97%	Excellent	95%	Easily rinsed	90	Good	Heavy corrosion	-0.055%
• Turco	T-2822	Homogeneous	Smooth, even coat	8:18	6:20	99%	99%	Excellent	>99%	Easily rinsed	91	Good	-1.36%	-0.950%
• Turco	T-6017	Slight separation	Fairly even coat	3:20	3:10	87%	98%	Excellent	>99%	Easily rinsed	92	Some lifting	7.5%	+0.258%

*Gloss on original paint film: 94.

** Maximum allowable weight change: aluminum ±0.016%; steel ±0.010%.

***N.D. = Not determined.

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to completely cover the test area. A more quantitative removal power test, which determined the total area lifted in a specific time using a specific volume of remover, showed that two of the removers lifted the paint film in 30 min or less using a specific remover volume (Table 32). The paint lifting process with time, resulting from this removal power test, is shown for the control and these two removers in Fig. 29, 30, and 31. These removers, Sprazee (BASF Wyandotte Corp.) and T-5873 stripper (Turco Co.), also showed good rinsability, coating residue, and remover residue. The rinsability test evaluates the ease of removal of the spent remover and the loosened paint film. The amount of paint film removed after a 2-hr contact time is measured by the coating residue test as a percent of total area. The remover residue test determined the ease of rinsing remover from bare substrate after exposure to 100°F for 15 min. Sprazee and T-5873 strippers were selected from the non-acid brush-on strippers for further evaluation.

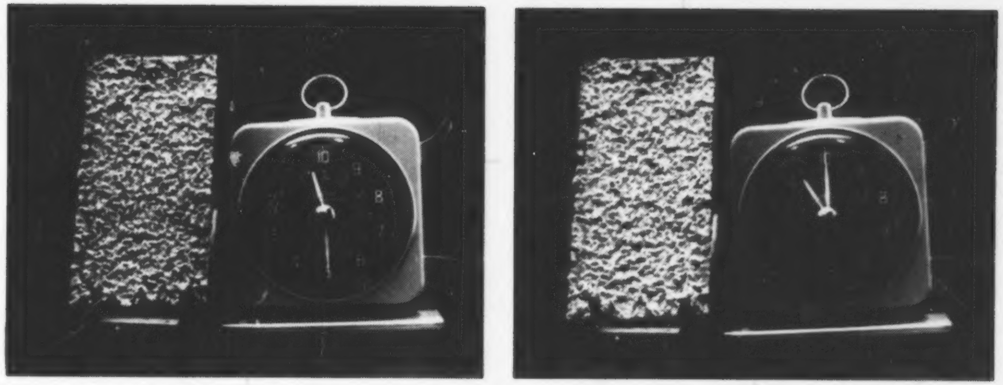
Each of the three candidate acid brush-on strippers (Oakite Visstrip, Turco T-2822, and Turco-6017) showed excellent removal power in both the time and area-lifted removal tests (Ref. Table 29). The paint lifting process with time in this removal power test is shown in Figures 32, 33, and 34 for these strippers. These strippers also showed excellent rinsability, coating residue, and remover residue properties. The removers were then subjected to further evaluation.

The refinishing properties of the selected brush-on strippers were determined by applying the paint system (epoxy primer per MIL-C-81773) to panels which had been stripped with each candidate system, and then testing to determine the adhesion, appearance, and gloss of the applied paint film (Ref. Table 30). The appearance of each of the refinished films was good, with a reduction in gloss from the original paint film of only 2 to 4 units. The adhesion of refinished films was determined by the wet tape test. Good adhesion was evident on all but one of the refinished films; in that one, which used the Turco T-6017 stripper, the topcoat lifted from the primer on one of the two panels; the other panel showed good adhesion. The corrosion compatibility of the selected paint strippers was also determined for aluminum and steel substrates. As expected, the acid paint strippers are not compatible with either aluminum or steel substrates. These substrates corroded to varying degrees after one week exposure to the paint stripper at 38°C (100°F). The Turco T-5873 non-acid stripper also showed corrosion of the steel substrate.

The performance of the candidate immersion-type paint strippers was evaluated in tests similar to those used for the brush-on type removers, with the major difference being the paint removal technique. Results are summarized in Table 33. One of the two non-acid immersion-type strippers evaluated (Magnus 766) showed good removal power and good rinsability, with 90% paint lifted after a 22-min contact time (Fig. 35). Two acid-immersion paint strippers were evaluated; one of these (Oakite Stripper SA) showed good removal power with excellent rinsability and a coating residue of only 2% after an 11-min contract time (Fig. 36). This stripper left a residue after contact with the bare substrate for 15 min at 38C (100°F) and thorough rinsing. The refinishing properties and metal compatibility of the selected immersion paint strippers were also evaluated. The refinishing properties of both strippers were good, with a 5-unit loss of gloss compared to the original paint film and only slight lifting of the paint system and small blisters in the wet tape test of the Stripper SA-stripped panels. The corrosion tests shows that the acid stripper was not compatible with either aluminum or steel, while the non-acid stripper (Magnus 766) was compatible only with the aluminum substrate (Ref. Fig. 35).

ECONOMIC AND ENVIRONMENTAL EVALUATION

Phenolic and non-phenolic paint strippers have basically the same requirements for paint removal and refinishing procedures. Although equipment requirements are the same, the time requirements are generally longer for non-phenolic strippers. Non-phenolic strippers do not pose any environmental hazards from accidental discharge of phenols, as do the phenolic strippers. Cost differences result from the disposal requirements to prevent environmental contamination.



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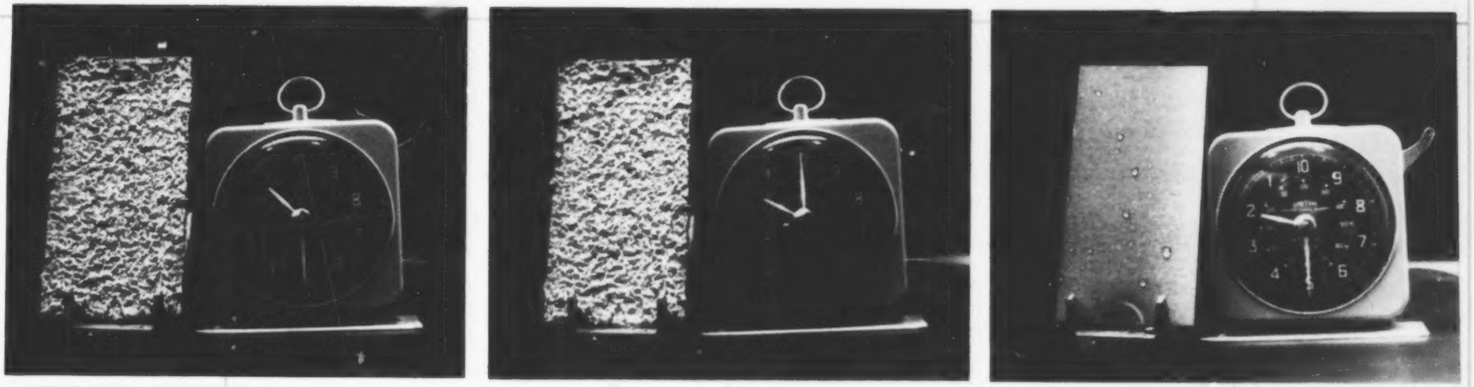

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Figure 29. Turco T-5469 Non-acid Brush-on Phenolic Paint Stripper (Control): Removal Power Tests.

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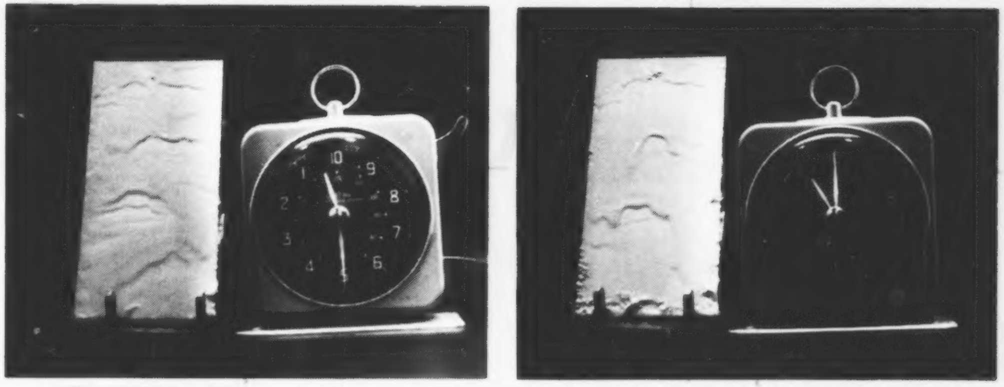

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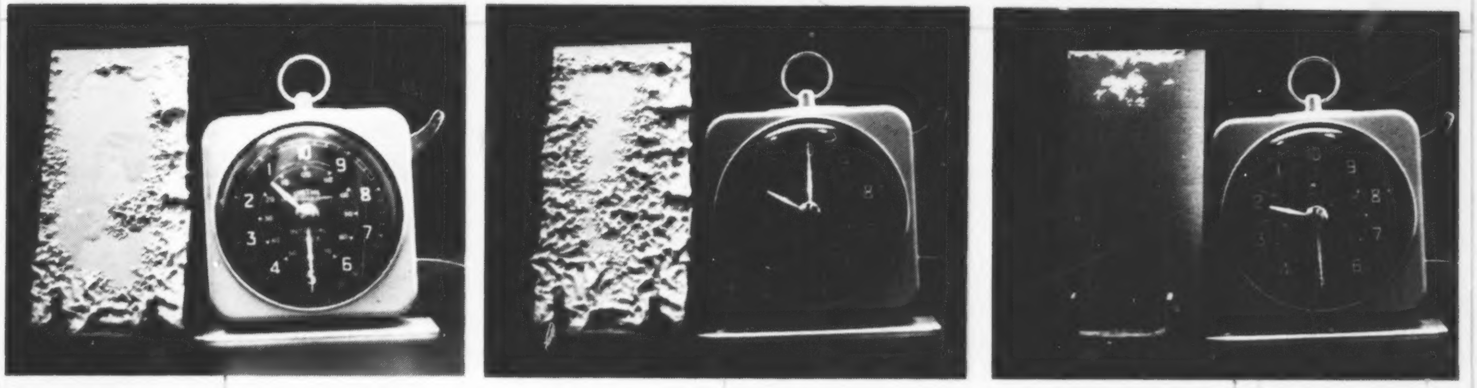

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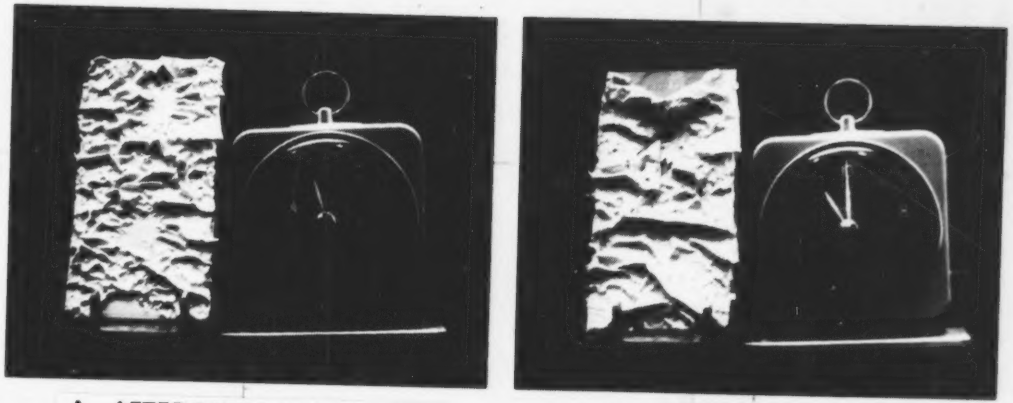
Figure 30. BASF Sprazee Non-acid Brush-on Paint Stripper: Removal Power Tests.



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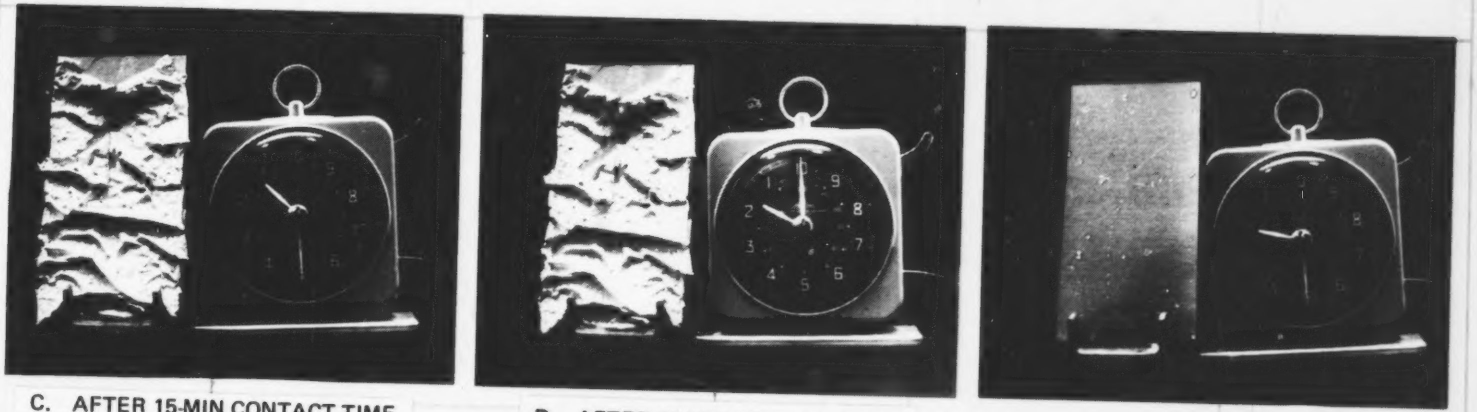

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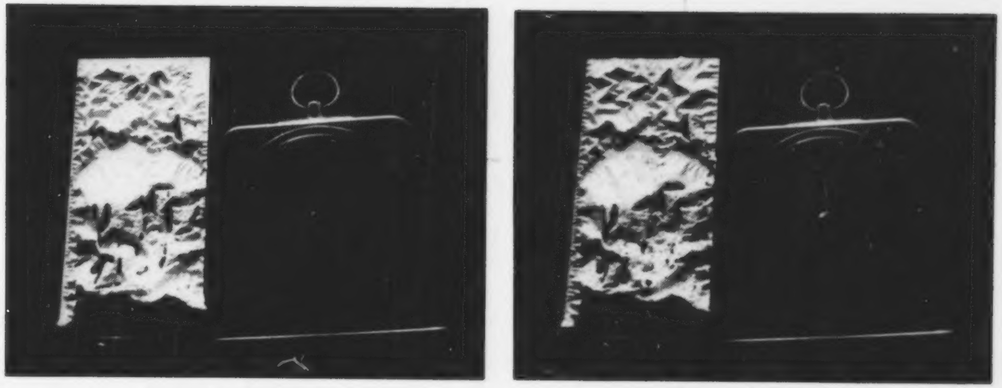
Figure 31. Turco T-5873 Non-acid Brush-on Paint Stripper: Removal Power Test.



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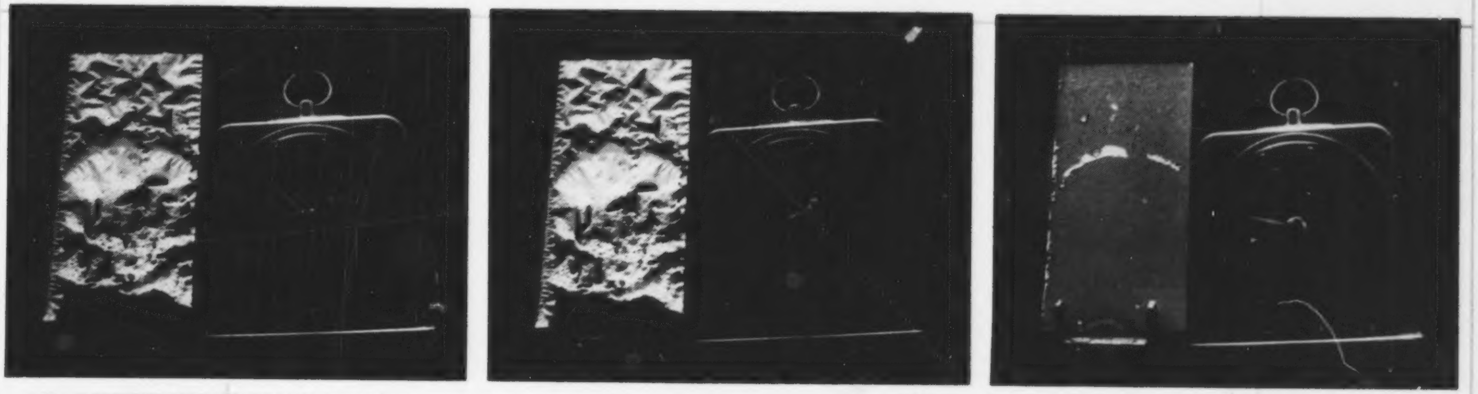

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Figure 32. Oakite Visstrip Acid Brush-on Paint Stripper: Removal Power Test.

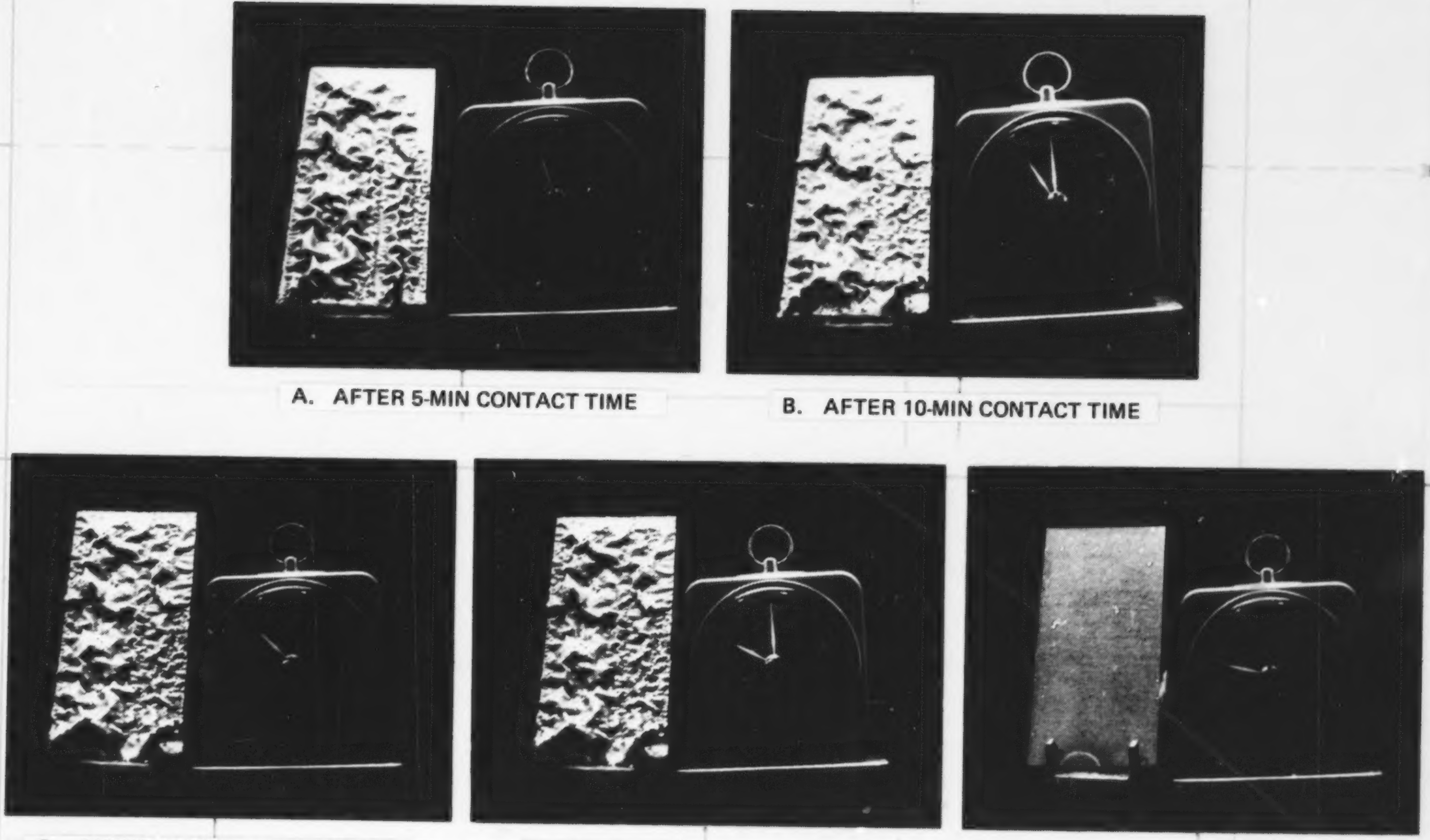
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Figure 33. Turco T-2822 Acid Brush-on Paint Stripper: Removal Power Test.

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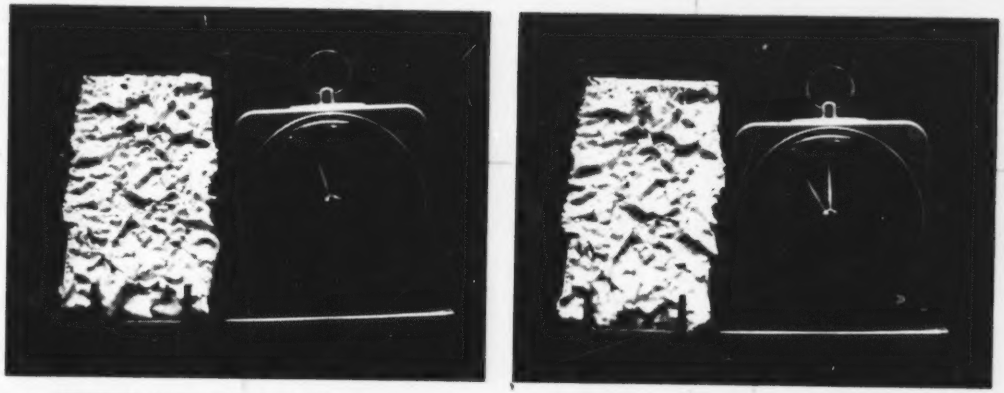

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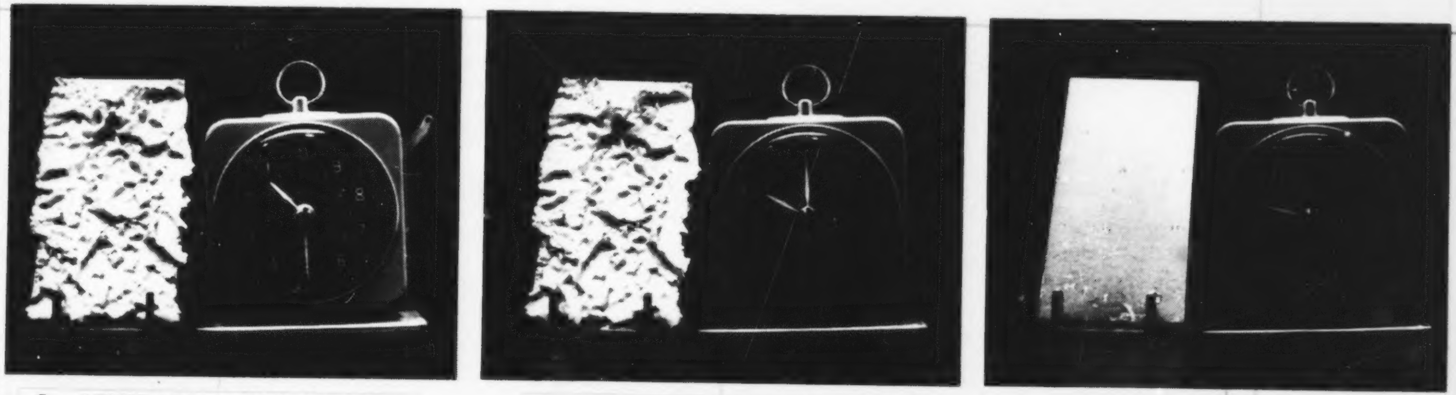

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Figure 34. Turco T-6017 Acid Brush-on Paint Stripper: Removal Power Test.

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TABLE 33. IMMERSION PAINT STRIPPERS: PRELIMINARY EVALUATION AND CHARACTERIZATION

Manufacturer	Trade name	Appearance	Removal power (time to lift, min: sec)	Rinsability	Coating residue (% removed)	Remover residue	Refinishing properties		Corrosion compatibility weight change, %**	
							Gloss*	Adhesion	Aluminum	Steel
Non-phenolic, non-acid										
● BASF	Rapoff	Separated	>80:00	Good	< 1%	Much residue	–	–	–	–
● Magnus	766	Slight separation	22:08	Good	90%	Some residue	89	Good	-0.003%	-0.112
Non-phenolic, acid										
● Oakite	Stripper EZ	Separated	>80:00	Fair	10%	Slight residue	–	–	–	–
● Oakite	Stripper SA	Homogeneous	11:08	Excellent	98%	Much residue	89	Sl lifting	-5.88%	-0.912

* Gloss on original paint film: 94

** Maximum allowable weight change: aluminum - ±0.016%; steel ±0.010%

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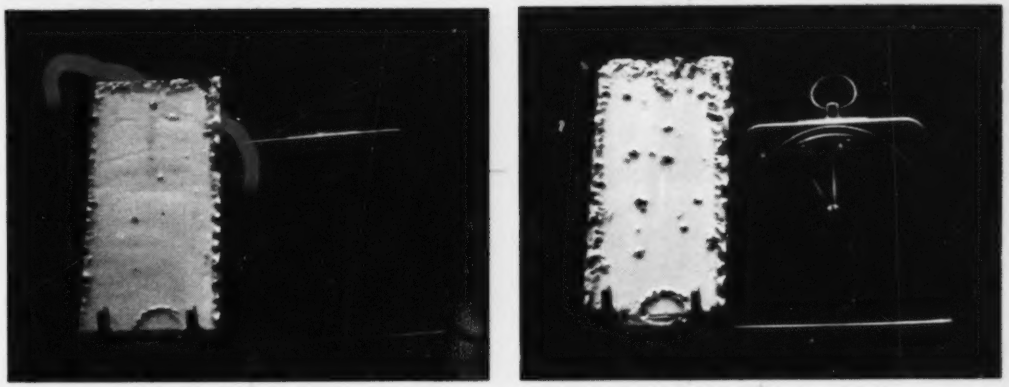

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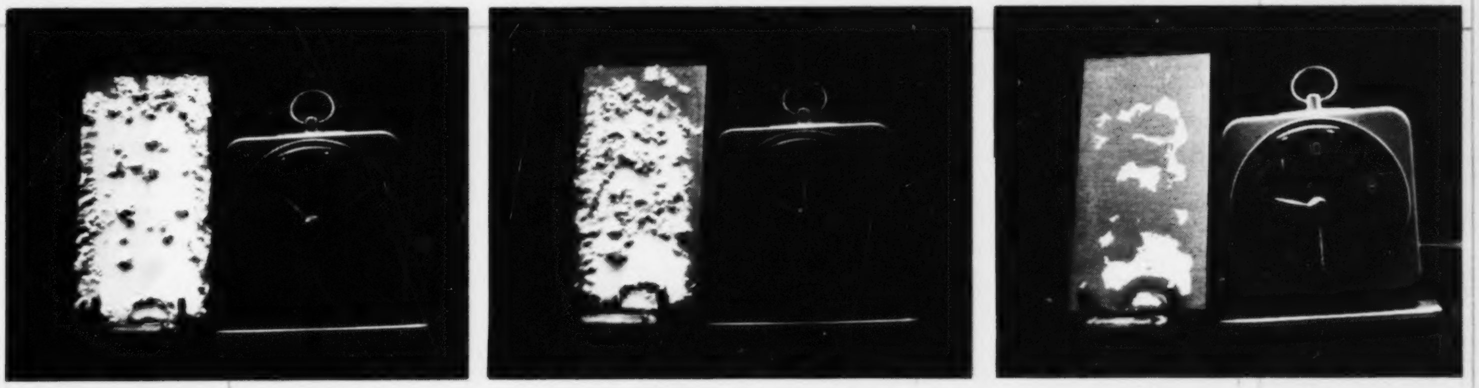

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Figure 35. Magnus 766 Non-acid Immersion Paint Stipper: Removal Power Test.

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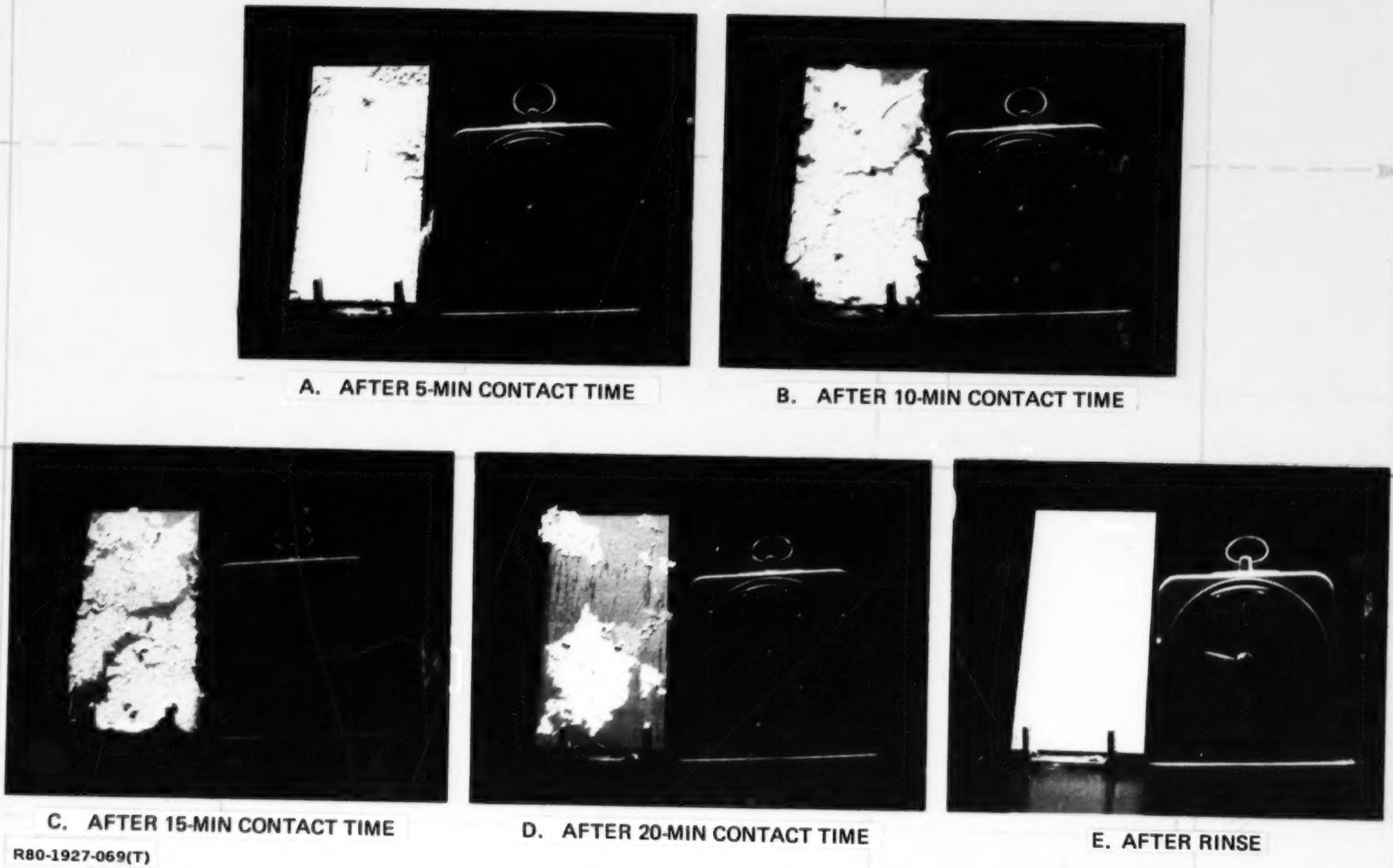

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Figure 36. Oakite Stripper SA Acid-Immersion Paint Stripper: Removal Power Test.

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3. Mock, M.A., "Water-Borne Coatings," *Materials Engineering*, August 1975.

4. O'Sullivan, D.A., "Trivalent Chromium is Basis of Plating Process," *Chemical and Engineering News*, June 1975.

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12. "Mechanical Plating," 3M Company Product Bulletin, 1975.

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APPENDIX A

TOXIC-EFFECT THRESHOLD-LEVEL TREATMENT PROBLEMS

TOXIC EFFECTS OF CYANIDES, METAL SOLUTIONS AND ORGANIC SOLVENTS ON HUMANS, ANIMALS AND PLANTS

Cyanides

Workers, such as electroplaters and picklers, who are daily exposed to cyanide solutions, may develop a "cyanide" rash, characterized by itching, and by macular, papular and vesicular erruptions. Frequently there is secondary infection. Exposure to small amounts of cyanide compounds over long periods of time is reported to cause loss of appetite, headache, weakness, nausea, dizziness, and symptoms of irritation of the upper respiratory tract and eyes.

Many cyanides evolve hydrocyanic acid rather easily. This is a flammable gas and is highly toxic. Carbon dioxide from the air is sufficiently acidic to liberate hydrocyanic acid from cyanide solutions.

Cyanide acts by inhibiting the phosphorylative oxidation reactions which permit cellular respiration. Many lower animals and fishes can convert a small amount of CN^- to thiocyanate ion, which does not inhibit respiratory enzyme activity. Cyanide compounds formed by the reaction of CN^- with heavy metals may be even more toxic.

Maximum acceptable concentrations of free cyanides in water are 1/20 (0.05) of the 96-hr LC_{50} value, determined using the receiving water in question and the most sensitive, important species in the area as test organisms in both static and flow-through boiassays. Maximum acceptable concentrations at any time or place are 0.005 milligram per liter (mg/l).

Cyanides in water derive their toxicity primarily from undissociated hydrogen cyanide (HCN) rather than from the cyanide ion (CN^-). HCN dissociates in water into H^+ and CN^- in a pH-dependent reaction. At a pH of 7 or below, less than one percent of the cyanide is present as CN^-; at a pH of 8, 6.7 percent; at a pH of 9, 42 percent; and at a pH of 10, 87 percent of the cyanide is dissasociated. There is more than a thousand-fold increase in toxicity of a nickel-cyanide complex associated with a decrease of pH from 8.0 to 6.5, and a pH change from 8.9 to 7.5 is said to increase the toxicity tenfold. The toxicity of cyanides increases with temperature and reductions in oxygen tensions. A temperature rise of 10°C produces a two- to three-fold increase in the rate of the lethal action of cyanide.

The median period of survival of trout at 17°C exposed to concentrations of 0.105 mg/l CN^- was reduced from eight hours at 95 percent oxygen saturation to 10 minutes at 45 percent oxygen saturation. Most of the literature on the toxicity of cyanides and hydrogen cyanide expresses toxicity in terms of the cyanide ion. It is reported that free cyanide concentrations from 0.05 to 0.10 mg/l as CN^- have proved fatal to many sensitive species. A level as low as 0.01 mg/l is known to have a pronounced, rapid, lasting effect on the swimming ability of salmon. Because safe HCN concentrations and acceptable LC_{50} application factors have not been positively demonstrated, conservative estimates of

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safe levels of cyanide in waters must be made. In determining acceptable cyanide levels for a given water body, both flow-through and static bioassays should be performed. These bioassays may reveal much greater toxicity than the flow-through method because the partial dissociation of some complex metallo-cyanide ions may be slow. On the other hand, volatile HCN may escape from a standing cyanide solution exposed to the atmosphere in static bioassay containers.

Cadmium

Cadmium is used in the production of copper, lead, silver and aluminum alloys. It also is used in metal finishing, ceramic manufacture and photography, and is a by-product of nuclear reactor operation. Salts of cadmium are used as insecticides and as anti-parasitic agents.

Cadmium may be removed from waste water as the metal hydroxide. Recovery of the metal by ion-exchange is sometimes economically attractive.

The permissible criterion for cadmium in public water supplies is 0.01 mg/l. Cadmium is especially dangerous, since it may combine synergistically with other toxic substances. Its principal effect upon aquatic life is the inhibition of bivalve shell production in mollusks.

Cadmium has been reported in concentrations as high as 3.2 mg/l in waters off Long Island. Such concentrations are the result of electroplating and chemical processing operations.

Cadmium toxicity has been implicated in serious cardiovascular diseases in man. Based upon current knowledge, agricultural waters should not contain more than 0.005 mg/l of cadmium in order to prevent cumulative toxicity in crops headed for the market.

Copper

Public water supplies may tolerate as a permissible criterion one mg/l of copper. A desirable crtierion of virtual absence has been expressed. In general, excess copper is highly toxic to algae, sea plants and invertebrates, but is only moderately toxic to mammals. The relationships between concentrations of different heavy metals in a water supply are extremely important in the determination of ultimate copper toxicity. Positive and negative synergistic effects make necessary the individual evaluation of each situation.

Kills of 35 to 100 percent of primitive fresh water plant forms were obtained with copper concentrations as low as 0.5 mg/l. Concentrations of copper above 0.1 mg/l were found to be toxic for oysters. Lobsters transferred to tanks lined with copper, after living in aluminum, stainless steel and iron tanks for two months, died within one day.

For agricultural waters, it appears that copper toxicities are more feed-related than water-related. It should be noted that copper toxicities have been observed at copper concentrations as low as 0.1 mg/l in plant nutrient waters. The maximum safe concentrations of trace ions in farmstead water is 1.0, 0.01 and 0.20 mg/l for copper, cadmium and cyanide, respectively.

Chromium

Chromic acid and its salts have a corrosive action on the skin and mucous membranes. The lesions are confined to the exposed parts, affecting the skin of the hands and forearms, and the mucous membranes of the nasal system. The characteristic lesion is a deep, penetrating ulcer which, for the most part, does not tend to suppurate and which is slow in healing. Chromate salts are recognized carcinogens of the lungs, nasal cavity and paranasal sinus.

Hexavalent compounds are considered to be more toxic than the trivalent. The maximum permissible chromium concentration for public water supplies is 0.05 mg/l.

Organic Solvents

Acetone and other ketones are narcotic in high concentrations. In industry, no injurious effects from its use have been reported, other than skin irritations resulting from defatting action, or headaches from prolonged inhalation.

Toluene at a level of 200 to 500 parts per million (ppm) can cause headache, nausea and impairment of coordination, and at higher levels, anemia, leucopenia and enlarged liver may result.

Ethylbenzene is an irritant to the skin and mucous membranes. A concentration of 0.1 percent of the vapor in air is an irritant to the types of humans and 0.2 percent is extremely irritating, causing dizziness, irritation of the nose and throat, and a sense of constriction of the chest.

Trichloroethylene inhalation causes narcosis and anesthesia. Fatilities following severe, acute exposure have been attributed to ventricular fibrillation resulting in cardiac failure.

Cellosolve, which is 2-ethoxy ethanol, is an eye irritant. Animal experiments have shown 18-24 hour exposure to 0.6 percent vapor may produce congestion and edema of the lungs and congestion of the kidneys.

Butyl Cellosolve, which is 20-butoxy ethanol, presents little danger from vapor inhalation due to its low volatility.

LAWS AFFECTING THE USE OF CADMIUM AND COPPER CYANIDE PLATING AND ORGANIC SOLVENT SYSTEMS

The Federal Water Pollution Control Act Amendments of 1972 provide that the Administrator of the EPA shall publish and revise, from time to time, water quality criteria. These criteria shall reflect the latest scientific knowledge on all identifiable effects of pollutants on human health, fish and aquatic life, plant life, wildlife, shorelines, and recreation. EPA-recommended maximum concentrations for cadmium, chromium, copper and cyanide are presented in Table A-1.

TABLE A-1. EPA WATER QUALITY CRITERIA

Substance	Irrigation	Live stock	Fresh-water aquatic life	Public water supply	Marine water aquatic life
Cadmium	0.01 (continuous) 0.05 (< 21 yr)	0.05	0.03 (hard water) 0.04 (soft water)	0.01	0.01
Chromium	0.10 (cont) 1.00 (<21 yr)	1.00	0.05	0.05	0.10
Copper	0.20 (continuous) 5.00 (<21 yr)	0.50	0.006 (soft water) 0.033 (hard water)	1.00	0.05
Cyanide			0.005	0.20	0.01

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The Walsh-Healy Act, covering Federal requirements for industries engaged in government contract work, was the first broad legislation to include numerical occupational health standards. The threshold limit values (TLVs) of the American Council of Governmental Industrial Hygienists (ACGIH) were included in the health requirements under this Act. Table A-2 is a listing of TLVs for some of the solvents previously discussed. These standards have also been adopted by the OSHA.

TABLE A-2. ADOPTED THRESHOLD LEVEL VALUES

Substance	Concentration	
	ppm	mg/m^3
Acetone	1000	2400
2–butanone (methylethyl ketone)	200	590
2–butoxyethanol (butyl cellosolve) – skin	50	240
Ethylbenzene	100	435
Toluene – (skin)	100	375
Trichloroethylene	100	535

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Severe air pollution in Los Angeles, California, has resulted in limitations in the amount of organic solvents that can be discharged into the atmosphere by users of such materials. These restrictions are described in Rule 66 of the Rules and Regulations of the Air Pollution Control District, County of Los Angeles. There is an especially great concern over photochemically reactive solvents, which are substances that undergo chemical reactions with atmospheric constituents in the presence of sunlight. The products of these reactions often contribute further hazardous contaminates to the environment.

TREATMENT PROBLEMS

Cyanide Waste Waters

Because of the great toxicity of cyanides, the elimination of cyanide compounds from waters produced in metal-finishing operations has been standard practice in larger installations. Cyanide can be destroyed by treatment with chlorine gas in an alkaline solution at room temperature. The reaction is:

$$2NaCN + 5Cl_2 + 12NaOH \rightarrow N_2 + 2NaCO_3 + 10NaCl + 6H_2O$$

Vigorous agitation is required during the chlorination process. Alkanity must be maintained to prevent the formation and evolution of poisonous cyanogen chloride.

Cyanide can also be destroyed by hypochlorites such as sodium hypochlorite (NaOCl), calcium hypochlorite [$Ca(OCl)_2$], or bleaching powder ($CaOCl_2$). An example of the probable reaction is as follows:

$$2NaCN + 5NaOCl + H_2O \rightarrow N_2 + 2NaHCO_3 + 5NaCl$$

This procedure is relatively simple and nonhazardous, when compared to chlorination.

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Cyanide may be converted to relatively non-toxic cyanate, using chlorine gas, hypochlorites or hydrogen peroxide. The reactions are:

$$NaCN + Cl_2 + 2NaOH \rightarrow NaCNO + 2NaCl + H_2O$$

$$NaCN + NaOCl \rightarrow NaCNO + NaCl$$

$$NaCN + H_2O_2 \rightarrow NaCNO + H_2O$$

Other methods have been used that are not as well known. All of them, however, introduce a chemical waste treatment of substantial cost into plating operations.

Chromium Wastes

Law requires the treatment of hexavalent chromium-bearing water wastes. This is done by lowering pH, adding sulfur dioxide or sodium metabisulfite to reduce the hexavalent chromium, and then elevating the pH with caustic to precipitate the trivalent chromium as a hydroxide. The following chemical equations apply to these reactions:

SO_2 Reduction

$$SO_2 + H_2 \rightarrow H_2SO_3$$

$$2H_2CrO_4 + 2H_2SO_3 \rightarrow Cr_2(SO_4)_3 + 5H_2O$$

$Na_2S_2O_5$ Reduction

$$Na_2S_2O_5 + H_2O \rightarrow 2NaHSO_3$$

$$2H_2CrO_4 + 3NaHSO_3 + 3H_2SO_4 \rightarrow Cr_2(SO_4)_3 + 3NaHSO_4 + 5H_2O$$

Neutralization

$$Cr_2(SO_4)_3 + 3Ca(OH)_2 \rightarrow 2Cr(OH)_3 + 3CaSO_4$$

$$Cr_2(SO_4)_3 + 6NaOH \rightarrow 2Cr(OH)_3 + 3Na_2SO_4$$

$$Cr_2(SO_4)_3 + 3Na_2CO_3 + 3H_2O \rightarrow 2Cr(OH)_3 + 3Na_2SO_4 + 3CO_2$$

ENERGY CRISIS EFFECT ON PRESENT SYSTEMS

In the opinion of certain environmentalists, the present rates of energy growth are unrealistic, environmentally damaging, and artificially induced. These rates cannot continue for long, and we should try to bring them under control.

Today we consume 15 times the amount of energy we did 100 years ago, although our population has only tripled in that time. In the last five years, the growth rate in the consumption of energy in all forms has been five percent per year. Growth has been particularly phenomenal in electrical energy, being seven percent annually in recent years. Projections based on that rate of growth call for a doubling of electric power production about every 10 years. Long-range projections have indicated that this growth will have to be curtailed, despite the use of nuclear fuels, because of an eventual shortage of land, water and fuel.

Water-borne coatings as a replacement for organic solvent coatings are certainly very attractive from the energy point of view. More than 2.5 billion pounds of solvents were consumed globally in industrial and trade coatings in 1973. The caloric value could alternatively be used to heat 300,000 homes annually in northern climates. In addition, their non-flammability and non-toxicity would require less fire-fighting equipment, and solvent recovery systems would be eliminated. The physical properties of water-based coatings, when compared to organic-solvent-based coatings, are different, however, and research and development would be needed with water-borne systems before a substitution could be made.

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APPENDIX B

SIGNIFICANT COMPANY CONTRIBUTIONS

The authors would like to thank the following companies for providing samples of their products for coating test panels and for providing technical assistance in support of this test program:

- Bee Chemical Co., 2700 E. 170th St, Landing, Ill 60438
- DeSoto, Inc., 1700 S. Mt. Prospect Rd, Des Plaines, Ill 60018
- Enthone, Inc., Box 1900, New Haven, Conn 06508
- Glidden-Durkee Div., SCM Corp., Third & Barn Sts, Reading, Pa 19603
- B.F. Goodrich Chemical Co., 6100 Oak Tree Blvd, Cleveland, O. 44131
- Harshaw Chemical Co., 1945 E. 97th St, Cleveland, O. 44106
- Harstan Chemical Co., 1247 38th St, Brooklyn, NY 11218
- Hi-Shear Corp., 2600 Skypark Dr, Torrance, Calif 90509
- Lea-Ronal Inc., 272 Buffalo Ave, Freeport, NY 11520
- LNP Corp., 412 King St, Malvern, Pa 19355
- LNP Corp., 412 King St, Malvern, Pa 19355
- MacDermid, Inc., 50 Brookside Rd, Box 671, Waterbury, Conn 06720
- McDonnell Douglas Corp., Box 516, St. Louis, Mo 63166
- M&T Chemicals, Inc., Rahway, NJ 07565
- Plastonics Inc., 230 Locust St, Hartford, Conn 06114
- Rilsan Corp., 139 Harristown Rd, Glen Rock, NJ 07452
- Rohm and Haas Co., Spring House, Pa 19477
- Shell Chemical Co., One Shell Plaza, Houston, Tx 77002
- Sterling Lacquer Manufacturing Co., 3150 Brannon Ave, St. Louis, Mo 63139

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- 3M Co., 367 Grove St, Saint Paul, Minn 55101
- Tiodize Co., Inc., 15701 Industry Ln, Huntington Beach, Calif 92649
- Union Carbide Corp., One University Plaza, Hackensack, NJ 07601
- US Paint, Lacquer, and Chemical Co., 2115 Singleton, St. Louis, Mo 63103
- D Aircraft Products Co., 1191 Hawk Circle, Anaheim, Calif 92807
- Adcoat Inc., 172 E. LaJolla St., Placentia, Calif 92670
- Turco Products, Division of Purex Corp., 24600 S. Main Street, Carson, Calif 90745
- The Polymer Corporation, Reading, Pa 19603
- Interrad Corporation, 65 Harvard Ave., Stamford, Conn 06904
- Emser Werke AG, Selnaustrasse 16, CH-8039, Zurich, Switzerland
- Deft Chemical Coatings, 17451 Von Karman Ave., Irvine, Calif 92714
- Oakite Products, So. Valley Road, Berkeley Heights, NJ 07922
- Magnus Division, Economics Laboratory, Inc., St. Paul, Minn 55102
- BASF Wyandotte Corporation, Wyandotte, Mich 48192
- Randolph Products Co., Calstadt, NJ 07072
- Empire Plating Corporation, 8800 Evarts Road, Cleveland, Ohio 44104

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APPENDIX C

TEST METHODS

The tests conducted to demonstrate the viability of the systems evaluated under this program are shown in Tables C-1 and C-2. These tests (Federal, Military, and ASTM) are normally used as acceptance criteria by aerospace and metal-finishing firms.

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TABLE C-1. SELECTED ORGANIC SOLVENT REPLACEMENT COATINGS

Property	Procedure	Replacement coating for: Primer	Replacement coating for: Finish Coat
Surface appearance	Visual	✓	✓
Gloss	Methods 6101 & 6104		✓
Hiding power	Method 6121		✓
Loss of gloss	After 72 hr air dry		✓
Lifting	1,4 & 18 hr air dry — visual	✓	
Adhesion	Method 6301	✓	
Coating anchorage	Method 6304		✓
Anchorage	Water/rt/24 hr—tape test		✓
Flexibility	220°F/4 hr, cool rt, Method 6221		✓
Impact resistance	Method 6226		✓
Water resistance	Water/rt/18 hr—visual	✓	
Hydrolitic stability	200°F/95% rh/18 days—visual		✓
Lubricating oil resistance	Diester lube oil/250°F/4 hr		✓
Weather resistance	1 yr exposure at Fire Island, N.Y.		✓
Accelerated weathering	168 hr/Method 6151		✓
Resistance to heat	260°F/20 hr plus 325°F/1 hr		✓
Resistance to taping	Tape removal—visual		✓
Hydraulic fluid resistance	168 hr/rt — visual		✓
Coolant resistance	168 hr/rt — visual		✓
Skydrol 500 resistance	168 hr/rt — visual		✓
JP-5 fuel resistance	168 hr/rt — visual		✓
Temperature/humidity cycling	MIL-E-5275		✓

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TABLE C-2. SELECTED ELECTRODEPOSITION REPLACEMENT COATINGS

Property	Procedures per Federal Spec QQ-P-416C & MIL Spec. C-14550A	Replacement coating for:		
		Copper cyanide plating	Cadmium cyanide plating	Hexavalent cyanide plating
Thickness	Microscopic	✓	✓	✓
Adhesion	Sheet bend	✓	✓	✓
Corrosion	5% salt spray/168 hr	✓	✓	✓
Decarb. protection	H. T. steel sheet bend	✓	✓	✓
Hydrogen embrittlement	75% units/200 hr	✓	✓	✓
Solderability	Solder 450°F — bend test	✓		

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